COMPARATIVE EVALUATION OF THE SHEAR BOND STRENGTH OF THE BOND BETWEEN CERAMIC AND ENAMEL PRETREATED WITH DIFFERENT ETCHING METHODS – AN IN VITRO STUDY

Dissertation Submitted to

THE TAMILNADU DR. M.G.R. MEDICAL UNIVERSITY

In partial fulfillment for the Degree of

MASTER OF DENTAL SURGERY

BRANCH I

PROSTHODONTICS AND CROWN & BRIDGE

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CERTIFICATE

This is to certify that the dissertation titled "**COMPARATIVE EVALUATION OF THE SHEAR BOND STRENGTH OF THE BOND BETWEEN CERAMIC AND ENAMEL PRETREATED WITH DIFFERENT ETCHING METHODS – AN IN VITRO STUDY**" is a bonafide record work done by **Dr. ABDUL RAFE** under our guidance and to our satisfaction during his post graduate study period between 2009 – 2012.

This dissertation is submitted to **THE TAMILNADU DR. M.G.R. MEDICAL UNIVERSITY**, in partial fulfillment for the Degree of **MASTER OF DENTAL SURGERY – PROSTHODONTICS AND CROWN & BRIDGE, BRANCH I.** It has not been submitted (partial or full) for the award of any other degree or diploma.

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CERTIFICATE

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INTRODUCTION

The demand for esthetic restorations and for preservation of healthy tooth structure has lead to the development and improvement of esthetic restorative materials.[10] Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties, that simulate the appearance of natural dentition. Other desirable characteristics include translucence, fluorescence, chemical stability, biocompatibility, low thermal and electrical conductivity, compressive strength and a coefficient of thermal expansion similar to that of tooth structure. In spite of their many advantages, ceramics are fragile under tensile strain. This makes the ceramic susceptible to fracture during the luting procedure and under occlusal force.[8]

Metal-backed ceramics were developed with the objective of improving the mechanical properties of the overall restoration.[46] The ceramo-metal restoration, which combined the strength of metal with the esthetics of ceramic, improved the success of dental ceramics.[8] The presence of the metal sub-structure limits the optical properties of the ceramic due to reduced light transmission and the tendency for marginal discolouration that affects the esthetics.[14,46] Developments in dental-ceramic engineering have led to the introduction of new, commercially available systems that use a ceramic core to replace the metal framework.

The primary weakness of dental ceramics is their brittleness which is likely to be their most important clinical characteristic. The esthetic and biological advantages of ceramic restorations have led to many efforts to improve the mechanical properties of dental ceramics. Several strengthening techniques and principles were developed and has resulted in improved mechanical properties and heightened esthetics of dental ceramics.[21] These strengthened ceramics have been named as metal-free ceramics, or "All-ceramics" and have been indicated for inlays, onlays, crowns and fixed partial dentures.[8]

All-ceramic restorations have gained popularity in recent years for the restoration of anterior teeth due to their excellent esthetic quality, biocompatibility and fracture resistance. They also have low thermal and electrical conductance and a coefficient of thermal expansion that is similar to enamel and dentin, resulting in minimal marginal leakage.[29,35] These restorations offer superior esthetics compared with metal-ceramic restorations. The inherent brittleness of some ceramic materials, specific treatment modalities and certain clinical conditions require resin bonding of the completed ceramic restoration to the supporting tooth structures for long-term clinical success.[6]

The success of all-ceramic restorations depends in part on a durable bond being created between the hard tissues of the tooth and the adhesive cement. A durable bond between the adhesive cement and the restoration is

also critical throughout the lifetime of a restoration.[35] A strong durable resin bond provides high retention, improves marginal adaptation, prevents microleakage and increases the fracture resistance of the restored tooth and restoration.[6,38] The bonded all-ceramic restorations provide a successful esthetic and functional service for patients. Clinical studies show excellent long-term success of bonded ceramic restorations such as inlays, onlays, laminate veneers and crowns.[27]

Contemporary restorative dentistry places a definite emphasis on adhesion. Accordingly, a long-term survival of adhesive porcelain restorations depends on the success of a reliable bond between the porcelain, the composite luting agent and the dental substrates.[15,18]

The ceramic restorations require considerable support from the underlying luting agent and enamel/dentin in order to optimize the bond strength between the restorations and the natural tooth.[44,54] The durability and the clinical performance of bonded porcelain restorations are mainly due to the cementing agents and adhesive systems. The cementation procedure is one of the factors for the clinical success of ceramic restoration.[35,36] This includes optimum surface treatment of the ceramic as well as proper choice and manipulation of the luting agent.[27] Therefore, adequate ceramic surface conditioning is essential in order to have a strong resin bond that relies on the micromechanical interlocking and chemical bonding to the ceramic surface. Common treatment options for ceramic surface are grinding, abrasion with

diamond rotary instruments, airborne particle abrasion with aluminium oxide, acid etching and combinations of any of these methods.[7,48] Acid etching with solutions of hydrofluoric acid (HF) or ammonium bifluoride can achieve proper surface texture and roughness. Hydrofluoric acid solutions between 2.5% and 10% applied for 2 to 3 minutes seem to be most successful.[6] Silane coupling agent application improves the bond strength of porcelain to resin luting agent.[27]

The surface treatment of dental substrate prior to adhesive restorative procedures is an extremely important step of the bonding protocol and accounts for the clinical success of restorations. In the literature, various surface treatment methods like air abrasion, acid etching and laser irradiation have been shown to etch enamel/dentin for the ceramic bonded restorations.[4,19] Air abrasion is a technique that involves use of air pressure with aluminium oxide powders to abrade dental tissues and produce large rough, irregular surface areas.[32] This can be regarded as a form of macroetching. The air abraded surface (sand blasted) displayed obtuse angularities instead of the sharp irregularities of etched enamel surfaces which could lead to weak bond strengths.[4]

The chemical treatment of enamel was first proposed by Buonocore by etching the enamel surface with orthophosphoric acid and has been commonly used to increase the bond strength of bonded ceramic restorations.[19] The technique of etching with orthophosphoric acid is used to create an irregular

surface of enamel. This allows an increase in the prepared surface area available for the retention of the resin cement and an improvement in the marginal adaptation of all ceramic restorations. The retentive characteristics of acid conditioned enamel surfaces depend on the type of acid, etching time and chemical composition of the enamel. Acid etching contributes to micromechanical retention of the adhesive components between the restoration and the enamel. The disadvantage of acid etching is that demineralization of the enamel surface makes it more permeable and prone to long term acid attack and caries. Currently, the most widely used protocol for enamel etching is with 37% phosphoric acid for 15 seconds.[19,31,51]

Since the development of the ruby laser by Maiman in 1960, lasers have become widely used in medicine and dentistry. Technological advances during the last decade have resulted in the increased use of lasers in dentistry. Many of these advances have been directed at the use of lasers in clinical applications as an alternative to acid etching of enamel or dentin for bonding dental materials to the tooth surface.[26]

The CO_2 laser was the first dental laser approved by the US Food and Drug Administration (FDA) and has been successfully used in soft tissue surgeries. CO_2 lasers have been reported to alter enamel surfaces in such a way as to strengthen bonding of resin materials and these lased surfaces may be superior to acid-etched enamel surfaces. The Nd:YAG laser uses a fiber-optic delivery system that penetrates wet tissue more easily than the CO_2 laser.

Nd:YAG and ArF:excimer devices have been reported to engender a weaker bonding surface than can be achieved with acid etching. Other approved systems include the Er;Cr:YSGG laser and the Er:YAG laser. These systems can be used for both soft and hard tissue procedures.[30,50]

The Er:YAG laser, originally developed by Zharikov et al in 1975, was approved by the FDA in 1997 for removal of caries, cavity preparations and modification of dentin and enamel surfaces prior to restoring with adhesive restorations. The Er;Cr:YSGG laser system was investigated in 1995 by Eversole and Rizolu. This pulsed laser device, when used with an air-water spray, has cut enamel, dentin, cementum and bone efficiently and cleanly without creating a significant smear layer. This laser system has been designated as hydrokinetic system (HKS) and can be used for tooth preparation without causing deleterious pulpal effects.[10,30,50]

Laser etching has also become available as an alternative to acid etching of enamel and dentin. Laser irradiation in particular causes thermally induced changes in the enamel surfaces. It causes surface roughening and irregularity similar to those following acid etching. Laser etching is painless and does not involve either vibration or heat, making it highly attractive for routine use. Furthermore, laser etching of enamel has been reported to yield an anfractuous surface (fractured and uneven) and open dentinal tubules, both ideal for adhesion.[19]

The surface produced by laser irradiation is also acid resistant. Laser irradiation of the enamel modifies the calcium-phosphate ratio and leads to the formation of more stable and less acid-soluble compounds, thus reducing susceptibility to caries attack. Therefore laser etching of enamel might be advantageous over phosphoric acid etching.[50]

The use of both laser and acid together has also been reported to enhance the strength of bonding to hard tooth surfaces relative to those exposed to acid alone.[19]

The type of luting cement has an influence on the long term durability of bonded ceramic restorations. Since the use of all-ceramic restorations requires considerable support from underlying composite resin cement and enamel/dentin for a successful clinical outcome, the luting agent should have high bond strength, not only to the ceramic surface, but also to tooth structure. Resin cements have been selected for their advantageous mechanical and adhesive properties when compared with the conventional luting cements. The applications of dual-polymerizing resin cements for all-ceramic restorations have considerably increased due to the ability of these cements to polymerize completely and their greater resistance to occlusal loading.[28,35,37,39,47]

The international standards organization document, TR110405 Dental Materials-Guidance has recommended longer periods of storage in a solution may be necessary to determine durability of bonds.[28] The complex nature of the oral environment has a direct influence on the bond that is achieved

between the interfaces of bonded ceramic restoration especially of the cementing agent and hard tissue. Water absorption may reduce the mechanical properties of the resin based luting agents and is detrimental to the silane-ceramic bond.[33,42] Therefore, testing the samples following water storage is essential to better simulate the oral conditions and achieve predictable results.

The common tests used in literature for measuring the bond strength are three-point bending, tensile, microtensile and shear bond strength tests.[25] Shear strength testing is perhaps more clinically applicable because resistance to shear stresses are thought to be important in retaining restorations that have been bonded to enamel surfaces.[30] In this study, a conventional shear bond strength was used to evaluate the bond strength.

Studies that comparatively evaluate the shear bond strength between ceramic and enamel subjected to acid etching or irradiated with different laser systems are available.[19,30,32,50,51] However, research comparing the effects of Er;Cr:YSGG irradiated enamel with acid etched enamel on the shear bond strength with ceramic is sparse.[19,50,51] Also there are fewer studies comparing the combined effects of acid etching followed by laser etching with Er;Cr:YSGG laser system.

In light of the above, the aim of the present in vitro study was to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

The objectives of the present study included the following:

1. To evaluate the shear bond strength of the bond between ceramic and enamel pretreated with 37% phosphoric acid etching.
2. To evaluate the shear bond strength of the bond between ceramic and enamel pretreated with Er;Cr:YSGG laser etching.
3. To evaluate the shear bond strength of the bond between ceramic and enamel pretreated with a combination of 37% phosphoric acid etching followed by Er;Cr:YSGG laser etching.
4. To compare the shear bond strengths of the bond between ceramic bonded to acid etched enamel, laser etched enamel and a combination of acid and laser etched enamel.
5. To qualitatively analyse the surface topography of enamel pretreated with 37% phosphoric acid etching before ceramic bonding by scanning electron microscope (SEM) analysis.
6. To qualitatively analyse the surface topography of enamel pretreated with Er;Cr:YSGG laser etching before ceramic bonding by scanning electron microscope (SEM) analysis..
7. To qualitatively analyse the surface topography of enamel pretreated with a combination of 37% phosphoric acid etching followed by Er;Cr:YSGG laser etching before ceramic bonding by scanning electron microscope (SEM) analysis.

8. To qualitatively evaluate the mode of failure of debonded test sample of ceramic bonded to enamel pretreated with 37% phosphoric acid etching by scanning electron microscope (SEM) analysis.
9. To qualitatively evaluate the mode of failure of debonded test sample of ceramic bonded to enamel pretreated with Er;Cr:YSGG laser etching by scanning electron microscope (SEM) analysis.
10. To qualitatively evaluate the mode of failure of debonded test sample of ceramic bonded to enamel pretreated with a combination of 37% phosphoric acid etching followed by Er;Cr:YSGG laser etching by scanning electron microscope (SEM) analysis.

REVIEW OF LITERATURE

Stangel I et al (1987)[47] investigated the shear bond strength of composite resin to porcelain to optimize variables, of etching and use of type of composite, for bonding porcelain laminate veneers. Composite was bonded onto both etched and non etched porcelain using unfilled resin, silane and silane with dentin adhesive. The conclusion they derived was that porcelain etching significantly increased bond strength across all variables.

al Edris A et al (1990)[1] evaluated the etch patterns produced by 1) a combination of hydrofluoric acid, hydrochloric acid and nitric acid, 2) a combination of hydrofluoric acid and sulphuric acid and 3) acidulated phosphate fluoride gel on porcelain surface. They concluded based on the SEM analysis that hydrofluoric acid and its combinations produced the most similar etch patterns consistently and produced greater roughness which the authors concluded to mean greater retention.

Visuri SR et al (1996)[53] evaluated the shear bond strength of the composite bonded to Er:YAG laser prepared dentin. The authors used human extracted molars and the teeth were prepared to the dentinal surfaces with a laser or with a dental aerotor handpiece. From these samples a few of them were further surface treated by etching with acid while others were not before finally bonding with composite cylinders. The authors found that the laser

irradiated samples had improved bond strengths when compared with the other samples.

Brosh T et al (1997)[9] evaluated the effect of different combinations of surface treatments and bonding agents on the bond strength of repaired composites. Three hundred and sixty samples were split into six groups with one group serving as control and the other five were subjected to the following surface treatments 1) grinding with a diamond stone 2) sandblasting with microetcher 3) jet prophylaxis 4) grinding with green carborundum 5) hydrofluoric acid – etching (9%). The authors concluded that different combinations of surface treatments and bonding agents affect the bond strength with sandblasting surface treatment recording the highest value and hydrofluoric acid etching recording the lowest value of shear bond strength.

Chen JH et al (1998)[12] investigated the effect of different etching periods on the bond strength of a composite resin to porcelain. They used 5% hydrofluoric acid and examined different times of 0, 5, 30, 60, 120 and 180 seconds with sixteen samples in each group. The etched patterns created were observed under a scanning electron microscope and the bond strengths were tested under a universal testing machine. The authors concluded that etching porcelain for 120 seconds gives the highest bond strength.

Iwami Y et al (1998)[23] investigated the effect of the wetness of enamel and dentin surfaces on the shear bond strength of composites. After testing seven commercially available bonding systems with three different surface

preparations the authors concluded that some amount of saturation was necessary for dentin surfaces to obtain high bond strength. However the relative dryness or the wetness of the enamel surface had no bearing whatsoever on the bond strength as measured by the authors.

Lin S et al (1999)[30] assessed the shear bond strength of composite bonded to tooth structure treated with an Er;Cr:YSGG laser system and compared it to a surface treated with carbide burs. The teeth were prepared along their long axes and were cut into both the enamel and dentin. They were also divided into two subgroups of etched and non-etched. A SEM analysis of the laser prepared surface and the carbide bur prepared surface was also done revealing that laser prepared surface did not cause the formation of a smear layer and an almost similar topography for the bur prepared surface was observed. No significant differences were also observed by the authors across their groups.

Martinez-Insua A et al (2000)[31] evaluated the tensile bond strength of teeth treated with an Er:YAG laser and acid-etched teeth. Eighty healthy human premolars were used. Brackets were cemented to acid-etched enamel, laser-etched enamel, acid-etched dentin, or laser-etched dentin (20 teeth per group). Dentin was previously exposed using a high-speed handpiece. Acid-etching was with 37% orthophosphoric acid (15 seconds for enamel, 5 seconds for dentin). Laser etching was with Er:YAG laser (four 200 mJ pulses per second for enamel; four 160 mJ pulses per second for dentin). Brackets were

bonded with auto – curing resin paste, having first applied a primer (dentin only) and then light-cured bonding resin. The authors concluded that adhesion to dental hard tissues after Er:YAG laser etching is inferior to that obtained after conventional acid etching. Enamel and dentin surfaces prepared by Er:YAG laser etching show extensive subsurface fissuring that is unfavourable to adhesion.

Hara AT et al (2001)[22] evaluated the influence of different cross head speeds on shear bond strength test on the tooth surface using one hundred and twenty extracted bovine incisors, embedded in resin. According to the authors different cross head speeds influence the shear bond strength of the material being tested and its fracture pattern. They also advocated cross head speeds of 0.50 and 0.75 mm/min for obtaining accurate results.

Kitasako Y et al (2001)[28] studied the shear bond strengths of three resin cements to dentin over a period of three years in vitro. Ten bovine teeth were used each with three different materials Panvia21, BISTITE and MASA bond. The bond strengths were evaluated at 1 day, six months, one year and three years. The samples were stored in plain tap water at 37^{o} C, with the water being changed on a daily basis. In this study the authors found that MASA bond an auto polymerizing resin cement recorded the highest bond strengths throughout the time period but the bond strength of all the cements decreased progressively with the increase of time interval.

Shimada Y et al (2002)[44] evaluated the shear bond strength of dual-cured resin cement to glass ceramics after sandblasting, acid etching and silanation. A castable glass class ceramic with a crystalline phase was used as the substrate material. The glass surfaces, which were sandblasted, polished or etched with phosphoric acid or hydrofluoric acid and were subsequently bonded with a dual-cured resin cement both with and without a silane coupling agent. The authors concluded that a silane coupling agent mixed with an acidic primer can effectively increase the bonding strength between resin cement and cast glass ceramics.

Stewart GP et al (2002)[48] evaluated in vitro the shear bond strength of resin cements to both ceramic and dentin. The ceramic specimens received six different surface conditioning treatments: sanding with 600-grit silicon carbide paper, microetching with aluminium oxide, sanding followed by silane application, microetching followed by silane application, hydrofluoric acid-etching and hydrofluoric acid-etching. The authors concluded that bond strengths were highly dependent on surface conditioning with hydrofluoric acid etching followed by silane application emerging as the most effective and reliable method in their study.

Cura C et al (2003)[15] evaluated the shear bond strength of a luting composite to enamel with six different bonding systems. Seventy extracted human molars and premolars were used for the study onto whom ceramic discs were bonded with six commercially available bonding systems in groups

of ten each, with the last group serving as a control in which no bonding agent was used. The authors concluded that, though no significant variance was observed between the systems, the use of a bonding agent greatly increased the bond strength.

Khoroushi M et al (2003)[25] evaluated the effect of thermocycling on the shear bond strength of composite resin to porcelain. In this experimental study, forty porcelain blocks were prepared and randomly divided into four groups (n=10). All porcelain surfaces were etched with 9.6% hydrofluoric acid, rinsed and air dried. In two groups, silane pre-treatment was done. Composite-resin was subsequently added on the ceramic surfaces, and light-cured. A group each of specimens with the silane pre-treatment and without the silane pretreatment were then subjected to 1000 thermal cycles. The authors found that the shear bond strengths of sample decreased considerably after thermocycling.

Spohr AM et al (2003)[46] studied the influence of six different surface treatments on the tensile bond strength between a resin cement and ceramic, with and without the application of a silane coupling agent. The six methods studied were sandblasting (100 um) with no silanation, sandblasting (100 um) with silanation, sandblasting (50 um) with no silanation, sandblasting (50 um) with silanation, hydrofluoric acid etching with no silanation, hydrofluoric acid etching with silanation. The authors found that the use of a silane agent

improved the bond strength within the same groups and hydrofluoric acid etching recorded the highest bond strength values.

Usumez A et al (2003)[51] evaluated in vitro the bond strengths of porcelain laminate veneers to tooth surfaces prepared with acid and Er;Cr:YSGG laser conditioning. Three surface treatments were used: laser conditioning with Er;Cr:YSGG, 37% phosphoric acid, 10% maleic acid. The in vitro bond strengths of porcelain laminate veneers bonded to tooth surfaces that were laser etched showed results similar to orthophosphoric acid or maleic acid etched tooth surfaces.

Piwowarczyk A et al (2004)[37] investigated the in vitro shear bond strength of cementing agents to fixed prosthodontic restorative materials. High-gold-content alloy and high-strength aluminium oxide surfaces were airborne-particle–abraded, and pressable ceramics were hydrofluoric acid-etched and silanated prior to cementing. The cementing agents tested were a zinc-phosphate cement, glass ionomer cements, resin-modified glass ionomer cements and resin cements. The authors' findings indicated that resin cements exhibited strong bond strengths to specific prosthodontic materials.

Ramos RP et al (2004)[41] investigated the effect of Er:YAG laser on bonding to dentin and the interaction pattern of different adhesive systems with the lased substrate. Tensile bond strength of a self-etching and two total-etch systems to lased and non-lased dentin was evaluated and the adhesive interface morphology was examined by SEM. The authors concluded that

consistent hybrid layers were observed for conventionally treated specimens; whereas they were either absent or scarce hybridization zones were viewed for the lased subgroups.

Celik EU et al (2006)[10] evaluated the shear bond strength of different adhesives to Er:YAG laser prepared dentin. Seventy specimens obtained from 35 extracted human molars were embedded in polyester resin and ground with silicon carbide papers. The authors concluded that Er:YAG laser irradiation increased the shear bond strength to dentin.

Chimello-Sousa DT et al (2006)[13] evaluated the influence of Er:YAG laser irradiation on the bond strength of a restorative system on enamel, varying the irradiation distance. The samples were divided into six groups, with the first five being treated with Er:YAG laser with the irradiation distance at 11, 12, 14, 16 and 17 mm, while the last group served as the control and received treatment with phosphoric acid alone. The authors concluded that with increase in irradiation distance, the bond strength increased.

Souza-Gabriel AE et al (2006)[45] investigated the shear bond strength of rein modified glass ionomer cements to ER:YAG laser treated tooth structure. The authors found that the adhesion for enamel was more efficient than for dentin. The cavities prepared with a conventional bur (control group) presented higher bond strength values than those recorded for Er:YAG laser.

Kukiattrakoon B et al (2007)[29] studied the effect of different etching times of acidulated phosphate fluoride gel on the shear bond strength of high leucite ceramics bonded to composite resin. The authors concluded that there

was no significant difference in the bond strengths in either of the two different surface treatments when the times for APF use were between 7 to 10 minutes.

Duarte S et al (2009)[18] studied the effectiveness of immediate dentin sealing (IDS) on the marginal adaptation and tensile bond strength of total – etch and self etch adhesives. The authors used twenty recently extracted molars and standard MOD inlay preparations were made on them. The authors came to the conclusion that immediate dentin sealing greatly improves the bond strength when compared with conventional composite cementation technique.

Pekkan G et al (2009)[35] examined both the shear and tensile bond strengths between pressable ceramic and resin cements. Three commercially available dual polymerizing resin cements were used to bond the two different ceramic systems to a total of one hundred and twenty extracted human molar teeth. All the specimens were thermocycled before being sent for the tests. The authors state that cementing agents influence the bond to the hard tissue with the shear bond strength values being consistently and significantly higher than tensile bond strength values.

Ritter AV et al (2009)[42] evaluated the shear bond strengths of dual-cure composite luting agents used with dual-cure dental adhesives. The authors reported that on enamel, the total-etch adhesives performed better than their self-etch counterparts, while in dentin, the opposite was found, i.e., the

self-etch adhesives performed better than their total-etch counterparts. Thermocycling for 1800 cycles did not affect the shear bond strength of the materials tested to dentin and enamel.

Moslemi M et al (2010)[32] compared in vitro the shear bond strength of a fissure sealant to enamel penetrated with Er;Cr:YSGG laser or air abrasion followed by acid etching. The authors concluded that pretreatment of enamel surfaces with the Er;Cr:YSGG laser did not increase the effectiveness of conventional acid etching and subsequently the bond strength as opposed to pretreatment of the enamel surfaces with air abrasion

Qeblawi DM et al (2010)[39] studied the effect of zirconia surface treatment on the flexural strength and shear bond strength to a resin cement. The mechanical treatments used were: airborne particle abrasion, silicoating and wet hand grinding. The chemical treatments used were acid etching followed by silanation, silanation only, and application of zirconia primer. The authors concluded that a combination of mechanical and chemical conditioning of the zirconia surface was essential to develop a durable resin bond to zirconia.

Turkmen et al (2010)[50] evaluated the shear bond strength of composite bonded with three different adhesive systems to Er;Cr:YSGG laser prepared enamel. The bond strengths obtained were not significant between the non-etched and laser etched groups, however for the etched groups laser etching showed significantly higher bond strengths The authors concluded that

Er;Cr:YSGG laser-powered hydrokinetic system etched the enamel more effectively than 37% phosphoric acid.

Yuasa T et al (2010)[55] evaluated the effects of two years of storage on the shear bond strength of two self-etching adhesive systems studied. The authors concluded that both the self etching primer adhesive systems, produced adequate shear bond strength even after 2 years of storage and thermocycling between 5^{o} C and 55°C for 6000 cycles.

Dundar et al (2011)[19] evaluated the strength of the bond between porcelain laminate veneers and tooth surfaces etched with acid and laser, separately and together. The teeth studied comprised 60 incisors extracted for periodontal reasons. These were divided into four groups according to etching method: group 1, acid etching alone; group 2, acid etching followed by laser etching; group 3, laser etching followed by acid etching; group 4, laser etching alone. The teeth were etched with 37% phosphoric acid and an Er;Cr:YSGG laser system. After the shear tests, scanning electron microscopy images of the tooth surfaces were obtained at a magnification of ×3,800. Etching with acid alone yielded the highest mean value of bond shear strength (15.4±3.8 MPa), while laser etching followed by acid etching gave the lowest mean value (11.5±4.6 MPa). The mean values of the bond shear strength for acid etching followed by laser etching and laser etching alone were 13.8±3.9 MPa and 12.8±4.6 MPa, respectively. Statistical analysis revealed no significant differences between the groups.

MATERIALS AND METHODS

The present in vitro study was done to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

MATERIALS EMPLOYED:

1. 33 recently extracted maxillary central incisors (Fig.1)
2. Separating discs 0.7 mm thickness (Dentorium, New York, USA) (Fig.2)
3. Autopolymerizing clear acrylic resin (Cold cure, DPI-RR, India) (Fig.3)
4. Die lubricant (Yeti Dental, Germany) (Fig.4)
5. Inlay wax (GC Corporation, Tokyo, Japan) (Fig.5)
6. Sprue wax (Bego, Germany) (Fig.6)
7. Investment ring and crucible former (IPS Silicone Ring, Ivoclar Vivadent, Liechtenstein) (Fig.7)
8. Pattern sprue guide (IPS e.max Press Sprue Guide, Ivoclar Vivadent, Liechtenstein) (Fig.8)
9. Phosphate bonded investment material (Pressvest, Ivoclar Vivadent, Liechtenstein) (Fig.9)
10. Colloidal silica (Pressvest Liquid, Ivoclar Vivadent, Liechtenstein) (Fig.10)
11. Aluminum oxide powder 110 microns (Aluminox 110, Delta , India) (Fig.11)
12. Diamond discs (Edenta AG, Switzerland) (Fig.12)

13. Silicon carbide impregnated burs coarse (Dura Green, Shofu Dental, Japan) (Fig.13)
14. Silicon carbide impregnated burs fine (Dura White, Shofu Dental, Japan) (Fig.14)
15. Primer (Syntac Primer, Ivoclar Vivadent, Liechtenstein) (Fig.15a)
16. Adhesive (Syntac Adhesive, Ivoclar Vivadent, Liechtenstein) (Fig.15b)
17. Bonding agent (Heliobond, Ivoclar Vivadent, Liechtenstein) (Fig.15c)
18. Silane coupling agent (Monobond S, Ivoclar Vivadent, Liechtenstein) (Fig.15d)
19. Dual-cure resin luting cement (Variolink N, Ivoclar Vivadent, Liechtenstein) (Fig.16)

INSTRUMENTS AND EQUIPMENTS EMPLOYED:

1. P.K. Thomas wax up instruments (Dispodent, India) (Fig.17)
2. Aerotor hand piece (Pana air, NSK, Japan) (Fig.18)
3. Inverted cone diamond abrasive (Dia Burs, Mani, Germany) (Fig.19)
4. Flat end tapered diamond abrasive (Dia Burs, Mani, India) (Fig.20)
5. Light cure unit (Confident, India) (Fig.21)
6. Vacuum mixer (Whipmix, U.S.A) (Fig.22)
7. Burnout furnace (Technico, Technico Laboratory Products Pvt. Ltd., Chennai, India) (Fig.23)
8. Sandblaster (Delta, India) (Fig.24)
9. Incubator (Narang Industries Ltd., India) (Fig.25)
10. Universal testing machine (Lloyd instruments, Farnham, U.K.) (Fig.26)

11. Scanning electron microscope (SA400N, Canada) (Fig.27)
12. Custom-made stainless steel split mounting jig (for mounting teeth in acrylic) (Fig.28)
13. Custom-made stainless steel tooth preparation guide (Fig.29)
14. Custom-made stainless steel split mold (for fabricating wax blocks) (Fig.30)

Ceramic system employed:

1. Lithium disilicate ingots MO shade (IPS e.max Press, Ivoclar Vivadent, Liechtenstein) (Fig.31)
2. Boron nitride (IPS e.max Alox Plunger Separator, Ivoclar Vivadent, Liechtenstein) (Fig.32a)
3. Plunger (IPS e.max Alox Plunger, Ivoclar Vivadent, Liechtenstein) (Fig.32b)
4. Ceramic press furnace (Programat EP-3000, Ivoclar Vivadent, Liechtenstein) (Fig.33)
5. 7% Hydrofluoric acid gel (IPS Ceramic Etching Gel, Ivoclar Vivadent, Liechtenstein) (Fig.34)

Etching systems employed:

1. 37% Phosphoric acid etching gel (N-Etch, Ivoclar Vivadent, Liechtenstein) (Fig.35)
2. Er;Cr:YSGG Laser (Waterlase MD Turbo, Biolase Technology, United States of America) (Fig.36)

Description of custom-made stainless steel split mounting jig: (Fig.28)

In the present study, a custom-made stainless steel split jig (Fig.28) was fabricated to mount the extracted natural teeth. The dimensions of the jig were 75mm x 25mm x 25mm (l x b x h) with an inner open space of 25 mm x 15mm x 25mm (l x b x h), for embedding the tooth in acrylic resin blocks. The jig was sectioned into two exact halves along the length and the two parts were retained by screws. This was done to facilitate easy removal of the acrylic block with the embedded tooth and also for subsequent reseating during tooth preparation. The jig had four screw holes on the top to help in positioning and seating the custom-made tooth preparation guide onto its surface.

Description of custom-made stainless steel tooth preparation guide: (Fig.29)

In the present study, a custom-made stainless steel tooth preparation guide (Fig.29) measuring 65 mm x 50 mm x 5mm (l x b x h) with a central preparation box measuring 5mm x 5mm was fabricated. The custom-made tooth preparation guide had four screw holes through which it was secured onto the top of the mounting jig during tooth preparation. This metallic guide was used to prepare the natural tooth embedded in the acrylic block which was secured in the stainless steel split mounting jig.

Description of custom-made stainless steel split mold: (Fig.30)

In the present study, a custom-made stainless steel split mold (Fig.30) was fabricated to obtain the wax blocks. The dimension of the custom-made stainless steel split mold was used to fabricate wax blocks measuring 5x5 mm.

Its dimensions are 65 mm x 45 mm x 5 mm (l x b x h). The mold was also split along the center to help in easy retrieval of the wax blocks. The mold was first milled to size using a commercial lathe for its exterior dimensions. The interior dimensions of the mold space (5 x 5 mm) were obtained thereafter.

Description of Er;Cr:YSGG laser system: (Fig.36)

In the present in vitro study the laser system used for surface treatment was from Biolase Technology, USA and the model was Waterlase MD Turbo (Fig.36) which is an Er;Cr:YSGG (Erbium; Chromium: Yttrium, Scandium, Gallium, Garnett) laser having a wavelength of 2780 nm. The power settings can be adjusted from 0.1 W to 8.0 W as per the clinician's requirement and the procedure attempted. The pulse repetition rate of the system also offers an individual preference from 10 to 50 Hz pulses. The optical tips available can focus the laser beam to either a 500 or a 700 micron diameter depending on the size used. This laser acts by its absorbtion into the chormophores present in the target tissue namely hydroxyapatite and water. The chormophores upon absorbing the laser energy are caused to expand rapidly bringing about the action of ablation.

Description of universal testing machine: (Fig.26)

In the present study, the shear bond strength between ceramic and surface treated enamel was determined with the universal mechanical testing machine (Lloyd Instruments, Farnham, U.K.) (Fig.26). It consists of a lower chamber, upper chamber, a display board to display the amount of force needed and a computer. The upper member houses the hydraulic pressure

machine. The lower portion has a bench vice test specimen fixture to hold the test specimens. The whole unit is attached to a computer for recording and converting data as required.

Description of the Scanning Electron Microscope: (Fig.27)

In the present study, the surface of the test samples was analyzed qualitatively using a Scanning Electron Microscope (SEM) (SA400N, Canada) (Fig.27). The SEM uses a beam of highly energetic electrons to examine objects on a very fine scale. They reveal the fine structure of variety of materials. SEM uses a scanned beam instead of a fixed beam, and it is used primarily for the examination of thick samples through which light cannot pass. The specimens to be magnified may have some conductivity and may get charged up. Hence they are coated with a platinum layer to prevent the charging up and in order to increase the secondary emissions. Additional sputter coating with gold produces high contrast and resolution, while also increasing the signal/noise ratio of the coated samples

METHODOLOGY

The following methodology was adopted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

I. Selection of teeth

II. Placement of teeth in custom-made jig

III. Preparation of teeth

IV. Fabrication of ceramic blocks

   a. Preparation of wax blocks

   b. Spruing of wax blocks

   c. Investing the blocks

   d. Burnout procedure for wax blocks

   e. Pressing of ceramic

   f. Divesting and finishing of ceramic blocks

   g. Preparation of ceramic blocks for bonding

V. Grouping of prepared teeth for etching procedures

VI. Etching of prepared teeth surfaces

   a. Group A: samples were acid etched with 37% phosphoric acid

   b. Group B: samples were laser etched with Er;Cr:YSGG laser system

   c. Group C: samples were acid etched with 37% phosphoric acid followed by etching with Er;Cr:YSGG laser system

VII. Cementation of ceramic blocks

VIII. Aging of test samples

IX. Shear bond strength test for test samples

X. Statistical analysis

XI. Qualitative analysis of the surface topography of surface treated, prepared teeth samples before bonding with ceramic blocks

XII. Qualitative analysis of the cemented test samples after debonding

I. Selection of teeth (Fig.37a, b, c)

Thirty three freshly extracted maxillary central incisors (Fig.37a) were utilised for the study which were free of caries, fractures, and restorations. The crown lengths were measured from the cemento-enamel junction to the incisal edge and from the mesial line angle to the distal line angle. A minimum length of 12 mm and a width of 10 mm were maintained for all the specimens. The selected teeth were sectioned (Fig.37c) at 2 mm below their cementoenamel junction using a separating disc (Dentorium, New York, USA) (Fig.2). While sectioning the teeth, care was taken that the teeth were kept moist. On the palatal surface of the crowns two longitudinal 2 mm deep grooves of 1 mm width were made to aid in the retention of the sectioned crowns with the acrylic.

II. Placement of teeth in custom made stainless steel split jig: (Fig.38a, b)

The inner surfaces of both halves of the custom made stainless split jig were then coated uniformly with petroleum jelly and then screwed tightly into place. Autopolymerizing acrylic resin (Cold cure, DPI- RR, India) (Fig.3) was

then poured into the mold space till the top and the sectioned natural tooth was embedded into the acrylic resin (Fig.38a). The natural tooth was embedded in such a way that the labial surface was exposed, for tooth preparation. Once the excess was removed, the custom made stainless steel preparation guide was then placed over the custom made stainless steel split jig and then secured into place further ensuring that the crown was mounted correctly. Once the autopolymerizing resin (Cold cure, DPI-RR, India) (Fig.3) had sufficiently cured, the custom made stainless steel preparation guide was unscrewed and the custom made stainless steel split mold was separated by removing its screws and the acrylic block was retrieved (Fig.38b). The selected thirty three natural teeth were embedded into the acrylic resin in an identical manner.

III. Preparation of teeth: (Fig.39a, b, c, d, e)

The middle portion of the labial surface of the teeth was selected for the preparation because of its larger width. The acrylic block with the embedded tooth was positioned in the custom made stainless steel split mounting jig (Fig.28), and was secured tightly. The custom made stainless steel tooth preparation guide (Fig.29) was then placed on top and locked in place (Fig.39a). This enabled to make the preparation in the middle one third of the tooth with the guide.

Premarked inverted cone burs (Dia Burs, Mani, Germany) (Fig.19), were used with a 7 mm marking on their shanks (Fig.39a) measured from the tip to prepare through 2 mm into the enamel surface as the thickness of the metal preparation guide was 5 mm. Care was taken to limit the depth of the

preparation in accordance with the markings on the burs, so as to not extend into the dentin surface (Fig.39c). The preparation was done in order to simulate the clinical preparation of ceramic laminate veneer restoration. After accomplishing the general outline of the intended test sample (a 5 x 5 mm square with 2 mm depth) (Fig.39d) the area was marked and the tooth structure around this area was ground using a flat end tapered diamond abrasive (Dia Burs, Mani, Germany), to ensure no impedance during the test for shear bond strength (Fig.39e). In this manner a total of 33 teeth were prepared and randomly grouped as described later.

IV. Fabrication of ceramic blocks:

a. Preparation of wax blocks: (Fig.40a, b, c)

The custom made stainless steel split mold was lined with die lubricant (Yeti Dental, Germany) (Fig.4) on each side of the mold spaces to aid in the retrieval of the wax blocks. The wax custom made stainless steel split mold was then secured close and placed over a clean glass plate flush with its surface (Fig.40a). Inlay wax (GC Corporation, Tokyo, Japan) (Fig.5) was poured into the mold space in a molten state and was allowed to cool gradually at room temperature (Fig.40b). Then the mold was placed in a bowl of chilled water to further harden the wax blocks, for a minute. After this the mold was removed from the bowl and wiped dry. Before the screws on the split mold were removed the excess formed at the top was then gently carved out using a PKT no.4 instrument (Dispodent, India) (Fig.17). The resulting wax block

(measuring 5 x 5 mm) was then eased out with gentle finger pressure (Fig.40c). In this manner a total of 30 wax blocks were obtained.

b. Spruing of wax blocks: (Fig.41a, b)

The wax blocks were sprued using preformed sprue wax (Bego, Germany) (Fig.6) of 2 mm diameter. The sprued wax blocks were then attached onto the crucible former (Fig.40a) of the sili ring (IPS Silicone Ring, Ivoclar Vivadent, Liechtenstein) (Fig.7). They were then measured for distance and angulation using the manufacturers provided guide (IPS e.max Press Sprue Guide, Ivoclar Vivadent, Liechtenstein) (Fig.8) at an angle between 45^{o} and 60^{o} (Fig.41b). The sprued wax blocks with the crucible former were then placed inside the sili ring (IPS Silicone Ring, Ivoclar Vivadent, Liechtenstein) (Fig.7).

c. Investing the wax blocks: (Fig.42a, b, c, d)

The wax blocks were invested using graphite free, phosphate bonded investment material (Pressvest, Ivoclar Vivadent, Liechtenstein) (Fig.9). A 6 mm distance was provided between the wax blocks and top of the ring. As per the manufacturer's recommendation, 200 gm of phosphate bonded investment (Pressvest, Ivoclar Vivadent, Liechtenstein) (Fig.9) was mixed with 44ml of investment liquid which was prepared by mixing 26 ml of colloidal silica (Pressvest Liquid, Ivoclar Vivadent, Liechtenstein) (Fig.10) and 18 ml of distilled water. The investment powder and liquid were first hand mixed with a spatula until the entire material was wet thoroughly, followed by vacuum mixing using a vacuum mixer (Whipmix, U.S.A) (Fig.22)

for 60 seconds. Once the investment was mixed the entire block was painted with a thin layer of investment using a small brush (Fig.42a). The sili ring (IPS Silicone Ring, Ivoclar Vivadent, Liechtenstein) (Fig.7) was placed on the vibrator and the remainder of investment was vibrated slowly into the ring (IPS Silicone Ring, Ivoclar Vivadent, Liechtenstein) (Fig.42b). The excess investment was then removed (Fig.42c). The invested blocks were allowed to set for 60 minutes, and the sili ring was removed (Fig.42d).

d. Burnout procedure for the wax blocks: (Fig.43)

The invested molds were placed in a burnout furnace (Technico, Technico Laboratory Products Pvt. Ltd., Chennai, India) (Fig.23) after setting of the investment, for wax elimination. Investments with the wax blocks were left in the burnout furnace for a period of two and half hours. During the first hour, the temperature was raised from room temperature to 380°C; in the second hour, the temperature was raised to 900°C and during the last half hour the temperature was sustained at 900°C to accomplish complete burnout of the pattern without any residue. The investment mold was initially placed in the furnace (Technico, Technico Laboratory Products Pvt. Ltd., Chennai, India) (Fig.43) towards the rear wall, tipped with the opening facing down towards the floor of the furnace for the escape of molten material but not flush against it. The investment mold was reversed later near the end of the burnout cycle with the sprue hole facing upward to enable escape of the entrapped gases and also to allow oxygen contact to ensure complete burnout of the wax.

e. Pressing of ceramic samples: (Fig.44a, b ,c, d, e)

The investment mold was then carried to the press furnace (Programat EP-3000, Ivoclar Vivadent, Liechtenstein) (Fig.33) and placed on the centre of the mounting plate (Fig.44c). The selected ingot (IPS e.max Press, Ivoclar Vivadent, Liechtenstein) (Fig.31) was then loaded (Fig.44a) with the shade designation facing upward and after the plunger (IPS e.max Alox Plunger, Ivoclar Vivadent, Liechtenstein) (Fig.32b) was dipped in the plunger separator (IPS e.max Alox Plunger Separator, Ivoclar Vivadent, Liechtenstein) (Fig.32a) to avoid adherence to the investment material and it was placed upon the ingot (Fig.44b). The manufacturer's pre-set program for the mold size was selected and activated. The base temperature of which was at 700^{o} C, with a standby time of 6 minutes. The temperature rise was set to gradually increase to 920 ^{o} C over a period of 60 minutes, at which time the ingot was pressed into the mold. Following pressing, the mold was allowed to cool to room temperature (Fig.44d). It was then cut carefully (Fig.44e) to be divested subsequently.

f. Divesting and finishing of ceramic samples: (Fig.45a, b, c)

The remaining investment was slowly removed from the casting by sand blasting (Fig.45a) with 110µm alumina (Aluminox 110, Delta, India) (Fig.11) at 80 psi pressure in a sand blasting machine (Delta, India) (Fig.24). Sprues were sectioned (Fig.45b) using 0.7mm thin diamond discs (Edenta AG, Switzerland) (Fig.12). The sample was inspected under magnification for pressing defects. External surfaces were relieved of all nodules with a silicon carbide impregnated bur (Dura Green, Shofu Dental, Japan) (Fig.13) and

cleaned. This procedure was repeated for all thirty specimens. All the ceramic samples were finished (Fig.45c) using silicon carbide impregnated burs (Dura White, Shofu Dental, Japan) (Fig.14).

g. Preparation of ceramic blocks for bonding: (Fig.46)

The ceramic samples finished in the above manner were then placed against a marked glass plate to check for its flatness. The flat ceramic surface was etched with 7% hydrofluoric acid gel (IPS Ceramic Etching gel, Ivoclar Vivadent, Liechtenstein) (Fig.34) for 1 minute in order to condition the ceramic.

V. Grouping of prepared teeth for etching procedures:

The teeth were divided into three groups, namely, Group A, Group B and Group C and subjected to three different surface treatments, namely, acid etching, laser etching and a combination of acid etching followed by laser etching respectively.

VI. Etching of prepared teeth surfaces: (Fig.47a, b, c, d, e)

Group A – Acid etching with 37% phosphoric acid:

37% orthophosphoric acid (N Etch, Ivoclar Vivadent, Liechtenstein) (Fig.35) was injected onto the prepared enamel surface of the teeth in Group A (Fig.47a) and left for 15 seconds. The tooth surface was then washed with water under pressure using a two way syringe. Each surface was then dried using a chip blower only. The treated specimen (Fig.47c) was then kept aside carefully in a separate container to avoid contamination before bonding it to

the ceramic sample. In this manner a total of 11 teeth samples for Group A (n=11) were subjected to acid etching.

Group B – Laser etching with Er;Cr:YSGG laser system:

Er;Cr:YSGG laser system (Waterlase MD, Biolase, USA) (Fig.36) was used to ablate the prepared enamel surface of the teeth in Group B (Fig.47b). The distance between the tip of the device and the surface of the sectioned crown was kept at 1 mm, and the laser beam was applied to the entire surface for 20 seconds. The laser was applied at a wavelength of 2,780 nm with pulse duration of 140 μs and a repetition rate of 15 Hz. The laser energy was 75 mJ. Laser energy was delivered through a fibre-optic system via a sapphire tip terminal 600 μm in diameter and the surface was bathed with an adjustable air/water spray using a water level of 30% and an air level of 60%. The treated specimen (Fig.47d) was dried using a chip blower and then kept aside carefully in a separate container to prevent it from contamination before bonding it with the ceramic block. In this manner a total of 11 teeth samples for Group B (n=11) were subjected to laser etching.

Group C – Combination of acid etching with 37% phosphoric acid followed by laser etching with Er;Cr:YSGG laser system:

37% orthophosphoric acid (N Etch, Ivoclar Vivadent, Liechtenstein) (Fig.35) was injected onto the prepared surface of the teeth and left for 15 seconds. The teeth surface was then washed with water under pressure using a two way syringe. Each surface was then dried using a chip blower only. Er;Cr:YSGG laser system (Waterlase MD, Biolase, USA) (Fig.36) was used to

ablate the prepared surface of the tooth thereafter. The distance between the tip of the device and the surface of the sectioned crown was kept at 1 mm, and the laser beam was applied to the entire surface for 20 seconds. The laser was applied at a wavelength of 2,780 nm with pulse duration of 140 μs and a repetition rate of 15 Hz. The laser energy was 75 mJ. Laser energy was delivered through a fibre-optic system via a sapphire tip terminal 600 μm in diameter and the surface was bathed with an adjustable air/water spray using a water level of 30% and an air level of 60%. The treated specimen (Fig.47e) was then kept aside carefully in a separate container to prevent it from contamination before bonding it with the ceramic block. In this manner a total of 11 teeth samples for Group C (n=11) were subjected to a combination acid etching followed by laser etching.

One representative prepared tooth sample from each group (A,B and C) was randomly selected and set aside for the qualitative analysis of the surface topography of surface treated, prepared teeth samples before bonding with ceramic blocks. The remaining thirty pretreated teeth were kept for cementation procedures.

VII. Cementation of samples: (Fig.48a, b, c, d, e, f, g, h)

The silane coupling agent (Monobond S, Ivoclar Vivadent, Liechtenstein) (Fig.15d) was applied onto the previously etched ceramic block's bonding surface using a microbrush (Fig.48a) and left for 60 seconds and then air dried. The bonding agent (Heliobond, Ivoclar Vivadent, Liechtenstein) (Fig.15c) was then applied onto the silanated surface of the

ceramic block (Fig.48b) and then cured using a light cure unit (Confident, India) (Fig.21) according to the manufacturer's instructions.

The primer (Syntac Primer, Ivoclar Vivadent, Liechtenstein) (Fig.15a) was then applied onto the prepared tooth surface using a microbrush tip (Fig.48c) and left to dry for 20 seconds. Excess was then removed by blowing air using a chip blower. An adhesive (Syntac Adhesive, Ivoclar Vivadent, Liechtenstein) (Fig.15b) was then applied onto the prepared tooth surface using a microbrush tip (Fig.48d) and left to dry for 20 seconds. The excess was then removed by blowing air using a chip blower. The bonding agent (Heliobond, Ivoclar Vivadent, Liechtenstein) (Fig.15c) was then applied onto the tooth surface (Fig.48e) and then cured using a light cure unit (Confident, India) (Fig.21) according to the manufacturer's instructions. Equal amounts of the dual-cure resin luting cement's (Variolink N, Ivoclar Vivadent, Liechtenstein) (Fig.16) base and catalyst paste were then dispensed onto the mixing pad and mixed using a plastic spatula. The mixed cement was then applied onto the previously etched and silane treated surface of the ceramic block (Fig.48f) and then the ceramic block was then pressed against the tooth surface under light finger pressure (Fig.48g). The excess was carefully removed from the sides and the cement was further polymerized using a light cure unit (Confident, India) (Fig.21) for 40 seconds. In this manner thirty ceramic blocks were cemented to the enamel pretreated with three different etching methods (Fig.48h). They shall henceforth be referred to as test samples.

VIII. Aging of test samples: (Fig.49a, b)

The ceramic bonded to natural teeth test samples of Groups A,B and C were then stored in distilled water (Fig.49a) placed in an incubator (Fig.49b) (Narang Industries Ltd., India) (Fig.25) at 37^{O} C for seven days before testing them for their shear bond strengths. The water was changed on a daily basis. This was done to simulate the oral conditions.

IX. Shear bond strength test for test samples: (Fig.50)

The test samples were tested for shear bond strength using a universal testing machine (Lloyd Instruments, Farnham, United Kingdom) (Fig.26). The force was applied at 90^{O} to the long axis of the tooth. The acrylic mold was mounted in the lower member and the upper member had the chisel with a cross head. A shear force was applied to the ceramic test sample at a cross head speed of 0.5mm / min until fracture occurred (Fig.50). The maximum fracture loads were recorded in Newton. The recorded values were then divided by the surface area of the sample to obtain the shear bond strength values in MPa. A total of 30 test samples were tested in identical manner and the shear bond strengths were tabulated for statistical analysis.

X. Statistical analysis:

All the statistical tabulations were done using Microsoft Excel (Microsoft, U.S.A.). the SPSS (SPSS for Windows 10.05, SPSS Software Corporation, Munich, Germany) software package was used for statistical analysis. One-way ANOVA was used to compare the mean values of the three

groups (A, B, and C). Tukey-HSD was used as the post hoc test and a p value < 0.05 was considered statistically significant.

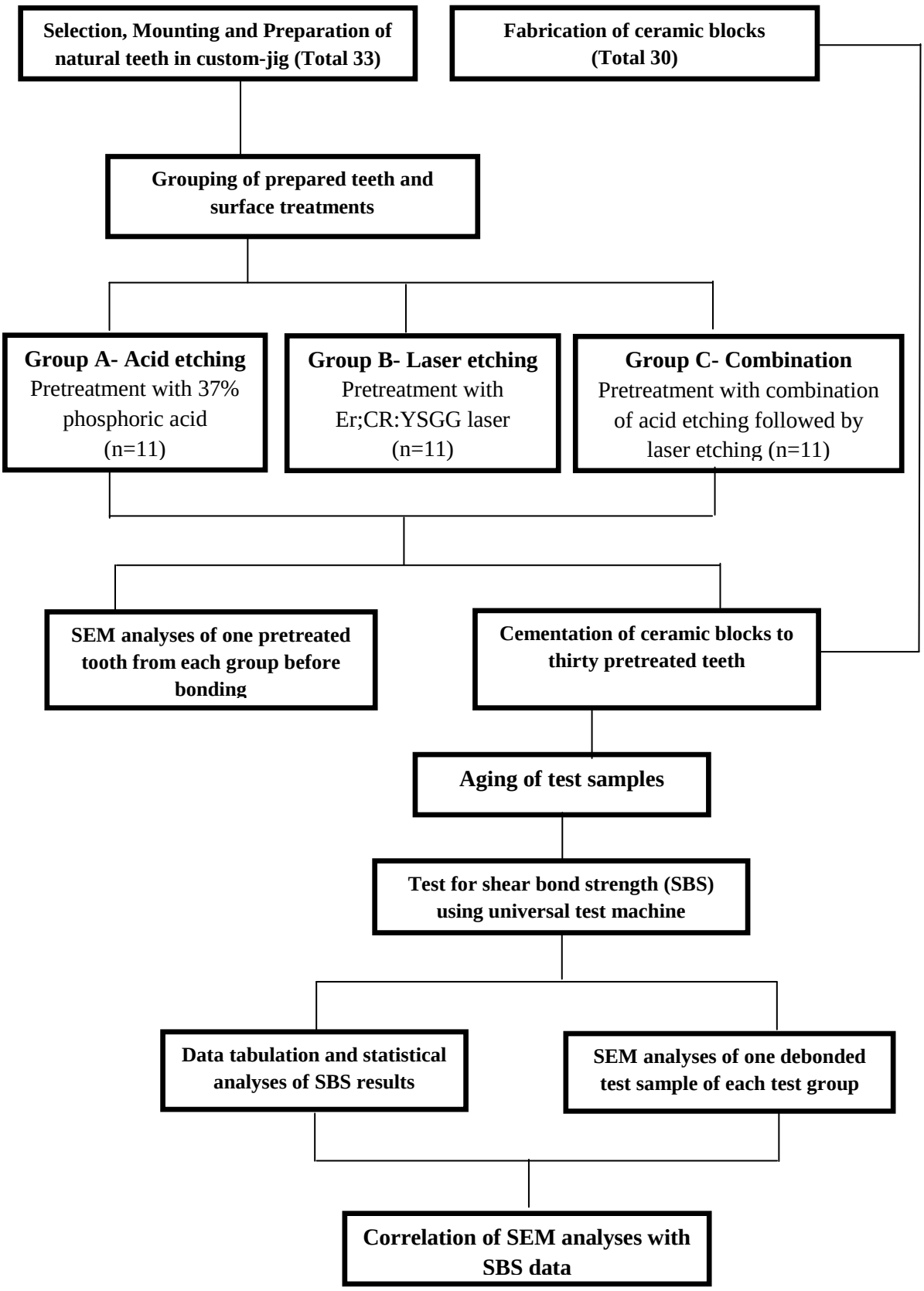
XI. Qualitative analysis of surface topography of surface treated, prepared teeth samples before bonding with ceramic blocks: (Fig.52)

SEM analysis was carried out on one representative surface treated, prepared teeth sample, randomly selected from each test group (Group A, Group B and Group C) before bonding of ceramic blocks using a scanning electron microscope (SA400N, Canada) (Fig.27). The samples were placed on stubs, secured in place with an adhesive tape and coated with a thin layer of gold in a gold sputtering system. Coated samples were examined under SEM to examine the surface topography of the treated samples at 10x, 500x and 1000x magnification (Fig.52).

XII. Qualitative analysis of cemented test samples after debonding: (Fig.53)

SEM analysis was carried to identify the mode of failure, on one representative tested sample from each test group (Group A, Group B and Group C) after debonding of ceramic blocks, using a scanning electron microscope (SA400N, Canada) (Fig.27). The samples were placed on stubs, secured in place with an adhesive tape and coated with a thin layer of gold in a gold sputtering system. Coated samples were examined under SEM to examine the mode of failure of the samples at 10x, 500x and 1000x magnifications (Fig.53).

METHODOLOGY-OVERVIEW



RESULTS

The present in vitro study was conducted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

Thirty three recently extracted central incisors were sectioned and mounted in acrylic using a custom-made stainless steel mounting jig. The teeth were prepared using a custom-made preparation guide limiting the depth of the preparation to the enamel. The teeth were divided into three groups, namely, Group A, Group B and Group C and subjected to three different surface treatments, namely, acid etching, laser etching and a combination of acid etching followed by laser etching respectively. One surface treated tooth from each test group was randomly selected for SEM analysis. A total of 30 pressed ceramic blocks were fabricated and bonded to the surface treated teeth. The test samples were subjected to aging and tested for shear bond strength in a universal testing machine until they debonded and the shear bond strength was calculated in MPa. The results obtained from this study were then subjected to statistical analysis. One debonded test sample from each test group was randomly selected for a qualitative assessment of the mode of failure by SEM analysis.

Table I shows basic values and mean value of shear bond strength for Group A test samples (acid etching).

Table II shows basic values and mean value of shear bond strength for Group B test samples (laser etching).

Table III shows basic values and mean value of shear bond strength for Group C test samples (combination of acid etching followed by laser etching).

Table IV shows the comparison between mean shear bond strength values of Group A (acid etching), Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using One-way ANOVA.

Table V shows the comparison between mean shear bond strength values of Group A (acid etching) and Group B (laser etching) test samples using Tukey HSD.

Table VI shows the comparison between mean shear bond strength values of Group A (acid etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD.

Table VII shows the comparison between mean shear bond strength values of Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD.

Graph I shows the basic values of shear bond strength of Group A test samples (acid etching).

Graph II shows the basic values of shear bond strength of Group B test samples (laser etching).

Graph III shows the basic values of shear bond strength of Group C test samples (combination of acid etching followed by laser etching).

Graph IV shows the comparison between mean values of shear bond strength values of Group A (acid etching), Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples.

Graph V shows the comparison between mean values of shear bond strength values of Group A (acid etching) and Group B (laser etching) test samples.

Graph VI shows the comparison between mean values of shear bond strength values of Group A (acid etching) and Group C (combination of acid etching followed by laser etching) test samples.

Graph VII shows the comparison between mean values of shear bond strength values of Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples.

Table I: Basic values and mean value of shear bond strength for Group A (acid etching) test samples

Sample no.	Shear Bond Strength
1	10.86
2	12.73
3	11.74
4	13.25
5	11.49
6	11.64
7	12.31
8	12.84
9	11.44
10	11.17
Mean	**11.9470**

Table II: **Basic values and mean value of shear bond strength for Group B (laser etching) test samples**

Sample No.	Shear Bond Strength
1	7.87
2	13.26
3	12.77
4	11.81
5	13.11
6	13.83
7	14.72
8	12.68
9	13.26
10	14.33
Mean	**12.7640**

Table III: **Basic values and mean value of shear bond strength for Group C (combination of acid etching followed by laser etching) test samples**

Sample no.	Shear Bond Strength
1	11.04
2	12.04
3	11.65
4	11.49
5	10.03
6	10.76
7	11.9
8	12.61
9	10.38
10	10.86
Mean	**11.2760**

Table IV: Comparison between mean shear bond strength values of Group A (acid etching), Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using One-way ANOVA

GROUP	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P–value
A	10	11.9470	+/-0.79081	0.049*
B	10	12.7640	+/-1.91180	
C	10	11.2760	+/-0.80199	

*P-value<0.05 denotes significance at the 5% level

Inference: On comparison between the mean shear bond strengths of Group A, Group B and Group C using One-way ANOVA it was found that there was a statistically significant difference between the mean shear bond strength of the three groups. Group B (laser etching) had the highest mean shear bond strength followed by Group A (acid etching) and the lowest shear bond strength value was observed in Group C (combination of acid etching followed by laser etching).

Table V: Comparison of mean shear bond strength values of Group A (acid etching) and Group B (laser etching) test samples using Tukey HSD test

GROUP	**Number of samples**	**Mean Shear Bond Strength** (MPa**)**	**Standard Deviation**	**P–value**
A	10	11.9470	+/-0.79081	0.342
B	10	12.7640	+/-1.91180	

P-value > 0.05; insignificant

Inference: On comparison between the mean shear bond strengths of Group A and Group B it was found that Group B had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, it was found that the p-value >0.05, denoting no statistically significant difference between these two groups.

Table VI: Comparison of mean shear bond strength values of Group A (acid etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD test

GROUP	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P–value
A	10	11.9470	+/-0.79081	0.480
C	10	11.2760	+/-.80119	

P-value > 0.05; insignificant

Inference: On comparison between the mean shear bond strengths of Group A and Group C it was found that Group A had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that the p-value >0.05, denoting no statistically significant difference between these two groups.

Table VII: Comparison of mean shear bond strength values of Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD test

GROUP	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P–value
B	10	12.7640	+/-1.91180	0.039*
C	10	11.2760	+/-0.80119	

*P-value < 0.05; significant at 5% level

Inference: On comparison between the mean shear bond strengths of Group B and Group C it was found that Group B had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that the p-value <0.05, denoting a statistically significant difference between these two groups.

Qualitative analysis of surface topography of pretreated enamel surfaces of Group A, Group B and Group C before bonding by scanning electron microscope under 10x, 500x and 1000x magnifications

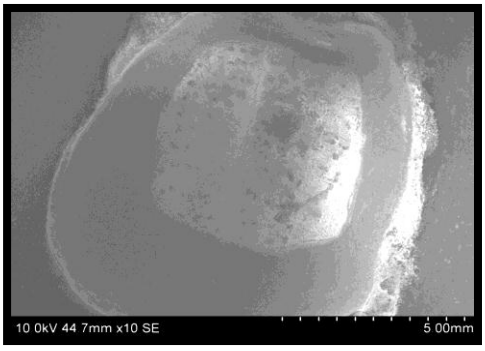


Fig.54 : Group A sample at 10x

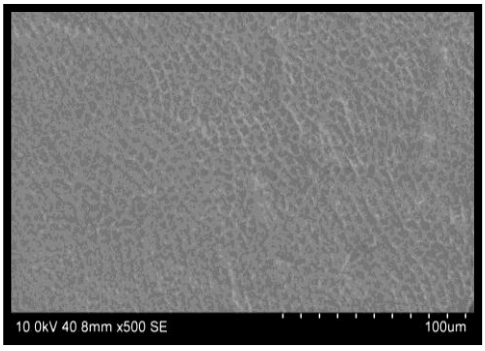


Fig.55 : Group A sample at 500x

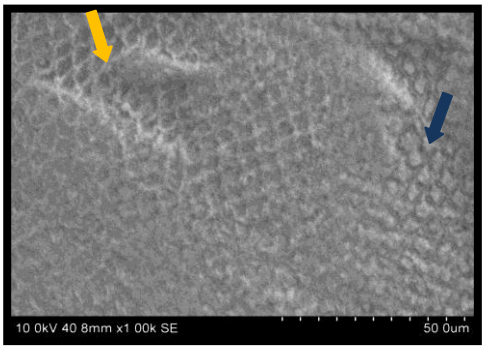


Fig.56 : Group A sample at 1000x

Group A inference :

The SEM photomicrograph of the Group A enamel sample, surface treated with 37% phosphoric acid shows a frosty appearance at a lower 10x magnification. Under 500x and 1000x magnifications it shows a uniformly microretentive surface with a clear presence of the key hole pattern of enamel. A random etching pattern of the enamel prisms corresponding to a type III pattern is visible: areas where the prism core is etched with the periphery intact (as indicated by the orange arrow) and areas where the core is intact and the peripheries are etched (as indicated by the blue arrow)

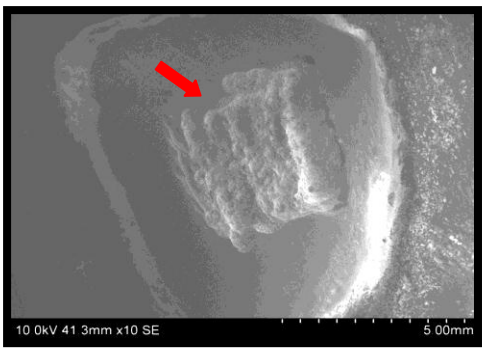


Fig.57: Group B sample at 10x

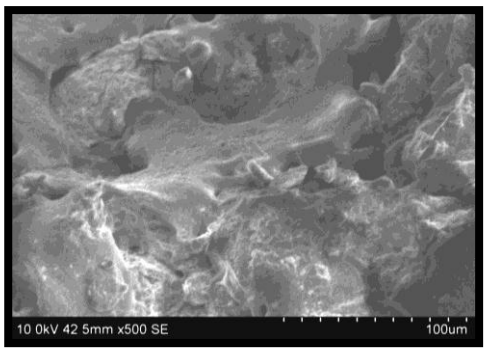


Fig.58: Group B sample at 500x

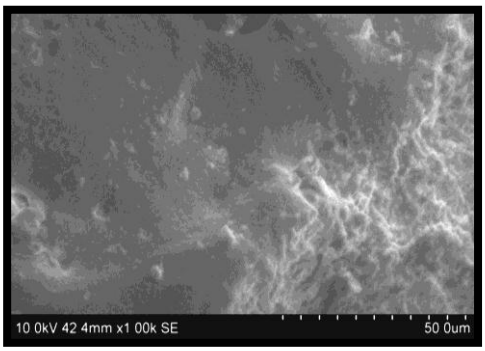


Fig.59 : Group B sample at 1000x

Group B inference:

The SEM photomicrograph of the Group B enamel sample, etched with Er;Cr:YSGG laser at a lower magnification of 10x shows a pronounced irregular surface with ridge-like elevations. There is a smoother untouched area (as indicated by red arrow). Under 500x and 1000x magnifications the lased surface shows slot-type pattern of enamel ablation indicating selective ablation of the enamel prisms over the lased surface. There is a definite microretentive surface with the presence of elevations and depressions of varying degrees. There was no recrystallization of enamel observed.

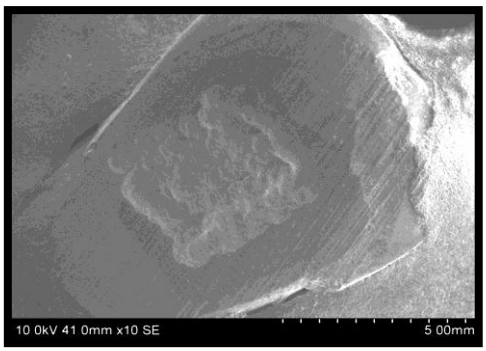


Fig.60: Group C sample at 10x

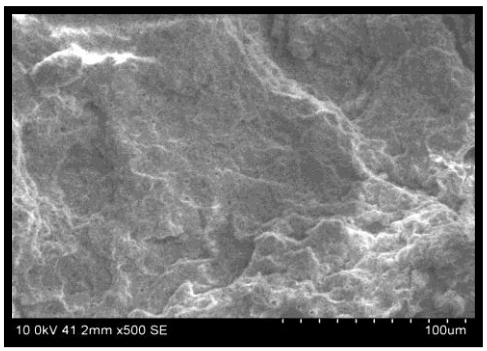


Fig.61: Group C sample at 500x

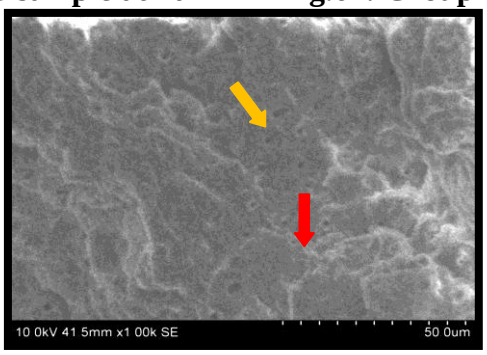


Fig.62: Group C sample at 1000x

Group C inference:

The SEM photomicrograph of the Group C enamel sample, surface treated with 37% phosphoric acid followed by Er;Cr:YSGG laser revealed a similar surface topography as seen with the Group B enamel sample, but to a much lesser extent at 10x magnification. Under 500x and 1000x magnifications a heterogeneous topography was revealed, showing both acid induced porosities (as indicated by orange arrow) and laser induced surface roughness (as indicated by red arrow). The elevations and depressions induced by laser ablation are far less pronounced as compared to that of Group B sample.

Qualitative analysis of the mode of failure of the debonded test samples of ceramic bonded to enamel of Group A, Group B and Group C by scanning electron microscope under 10x, 500x and 1000x magnifications

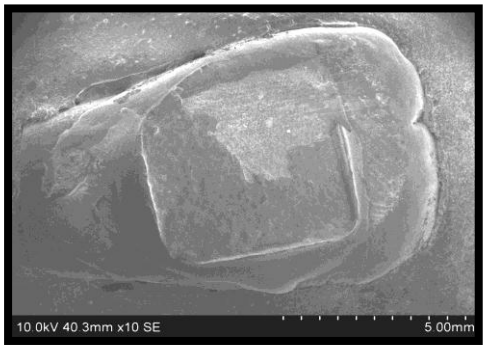


Fig.63 : Group A sample at 10x

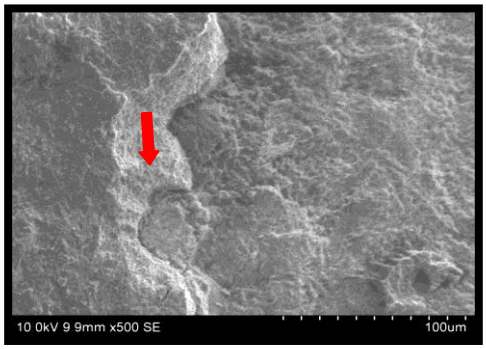


Fig.64 : Group A sample at 500x

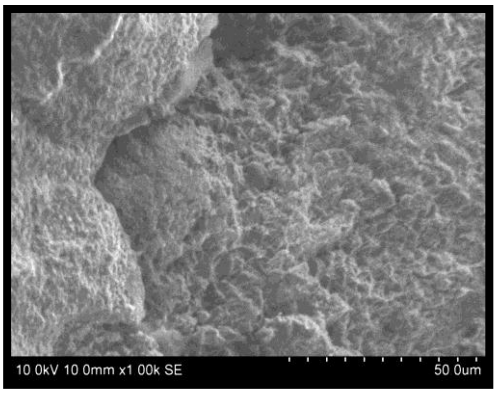


Fig.65 : Group A sample at 1000x

Group A Inference:

The SEM photomicrograph of the Group A sample at 10x magnification revealed a mixed failure pattern. There is a predominant cohesive failure within the cement and an adhesive pattern of failure between the enamel and cement. This can be clearly seen with the elevated cement layer (as indicated by red arrow) adjacent to the enamel layer at 500x and 1000x magnifications. The enamel layer shows the presence of prisms indicating areas of adhesive failure between enamel and the cement.

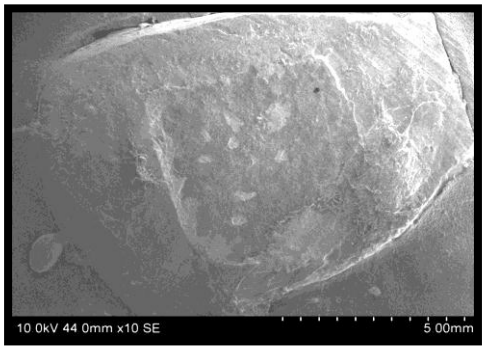


Fig.66: Group B sample at 10x

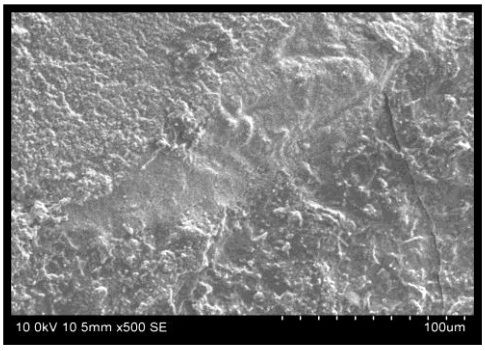


Fig.67: Group B sample at 500x

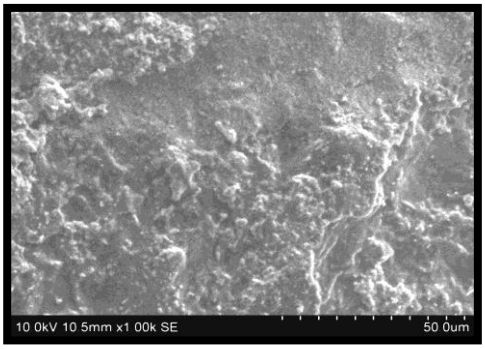


Fig.68: Group B sample at 1000x

Group B inference:

The SEM photomicrograph of the tested Group B sample at 10x magnification reveals a predominantly cohesive mode of failure in the cement with few areas of adhesive failure. Higher magnifications of 500x and 1000x reveal the clear presence of the cement layer.

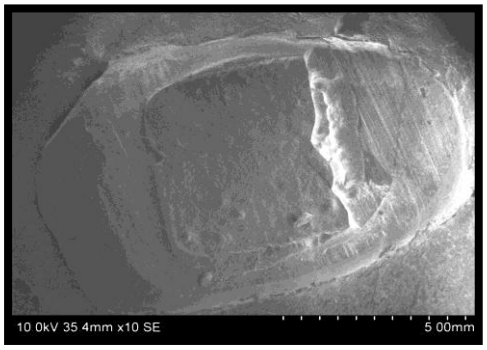


Fig.69: Group C sample at 10x

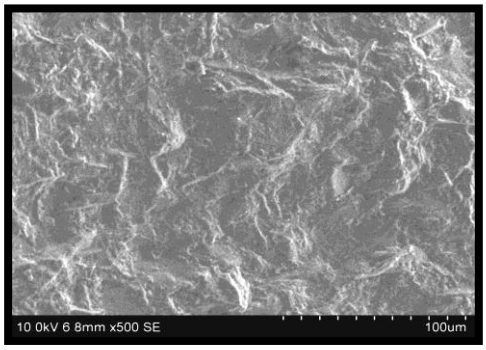


Fig.70 : Group C sample at 500x

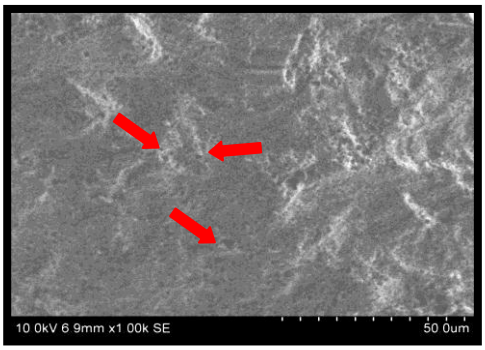


Fig.71 : Group C sample at 1000x

Group C inference:

The SEM photomicrograph of the tested Group C sample at 10x magnification reveals a predominantly adhesive failure pattern. At 500x and 1000x magnifications the surface topography correlated with the pretreatment surface topography showing the heterogenous enamel surface with mixed areas of laser etched enamel and acid induced porosities without the presence of resin tags (as indicated by orange arrow).

DISCUSSION

The present in vitro study was conducted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

Ceramics as dental materials have excellent physical, chemical and optical properties.[8,46] Since, they are brittle in nature the ceramo-metal restoration, was developed with the objective of improving the mechanical properties of the overall restoration; but the metal core affects the esthetics.[8,21,46] Several strengthening techniques were developed to improve the mechanical properties of dental ceramics and have resulted in the currently available all-ceramic systems.[16,21]

The popularity of all-ceramic restorations continues to grow due to their high esthetic quality and fracture resistance.[27,29] The success of all-ceramic restorations depends on the formation of a durable bond between the hard tissues of the tooth, adhesive cement and the restoration, as it helps to improve the fracture resistance of these restorations.[11,35] This includes optimum surface treatment of the ceramic, proper choice of luting agent and surface treatment of tooth.

Ceramic surface treatment is fundamental for bonding to resin.[6,8,27] The common surface treatments listed in literature are acid etching, airborne particle-abrasion, grinding and a combination of any of these methods.[9,27]

Acid etching of porcelain creates microporosities on the porcelain surface, which form a micromechanical interlock with the luting agent.[1] Several porcelain etchants have been developed like hydrofluoric acid and acidulated phosphate fluoride (APF).[1] The most commonly used etchant is a 10% solution of hydrofluoric acid.[1] Hydrofluoric acid attacks the glass phase of conventional ceramic materials producing a retentive surface for micromechanical bonding.[8,27] It has also been reported in literature that hydrofluoric acid solutions between 2.5% and 10% applied for one to four minutes are most successful in achieving a proper surface texture and roughness of ceramic surface.[27] In accordance with the literature available, the present study used a 7% hydrofluoric acid gel applied onto the ceramic blocks for one minute as the surface conditioning agent.

Recent developments in modern surface conditioning methods with silane coupling agents have resulted in improved bond strength of porcelain to the luting agent.[25,27] Silane application improves the wettability of the ceramic and contributes to covalent bond formation between the ceramic and the luting agent.[27,34] Silanes are bifunctional molecules that bond silicon dioxide with the OH groups on the ceramic surface and copolymerizes with the organic matrix of the resin cement.[27] It has also been reported that, etching and silanization significantly decreases microleakage.[6,25,27]

The treatment of dental substrate prior to adhesive restorative procedures is an extremely important step of the bonding protocol and

accounts for the clinical success of all-ceramic restorations.[13] During conventional tooth preparation with rotating instruments a smear layer is produced on the surface which consists mainly of pulverized enamel and dentin, carious debris, and bacteria.[10,19] The low surface energy of this layer prevents, impregnation of the enamel and dentin with the adhesive agent and thus, an adequate adhesion thereby affecting the durability of the bond between the restoration and the tooth.[10] The standard approach to solve this problem has been removal of the smear layer before sealing or bonding by surface treatment of the dental substrate.[10] The primary effect of enamel etching is to increase the surface area and thereby change the surface substrate from a low energy hydrophobic surface to a high-energy hydrophilic surface.[4,23]

In the literature, various surface treatments for treating enamel/dentin have been reported using chemicals like phosphoric acid, maleic acid and mechanical methods like intra oral air abrasion and laser etching.[4,5] Buonocore (1955), postulated that acids could be used to treat the prepared tooth surface before the application of resins.[10,15,31,55] The most widely used method is the application of 37% phosphoric acid for the enamel surface.[31] Phosphoric acid acts on the enamel by selectively dissolving the hydroxyapatite of the prisms, thereby facilitating penetration of the bonding agents and tag formation.[31] A disadvantage attributed to acid etching is that demineralisation of enamel surface makes it more permeable and prone to long term acid attack and

caries, especially if the demineralised substrate is not completely filled by the resin monomers.[12,13,31]

The other methods tried as alternatives to acid etching with phosphoric acid were other acids such as maleic acid,[51] or air abrasion using alumina 50 µm with/without acid etching[32] and laser etching.[4] Berk et al (2008)[4], in their in vitro study concluded that air abrasion was not a viable alternative to acid etching as it resulted in macroetching as opposed to microetching, attained with acid etching.[4] It has also been reported that etching with 37% phosphoric yields better bond strengths than etching with 10% maleic acid.[51] In accordance with the literature, the present in vitro study used 37% phosphoric acid to etch the enamel surfaces of the prepared tooth samples for 15 seconds.

In 1960, Theodore H. Maiman developed the method of light amplification by the stimulated emission of radiation, now commonly known by its acronym, LASER. Five years later Goldman et al investigated the application of lasers on hard dental tissues.[50] Advancements in laser technology have led to multiple dental applications such as soft tissue surgery, composite polymerization, tooth whitening, endodontic procedures and caries removal and cavity preparation with minimal pain and discomfort. Laser etching may be an alternative to acid etching of enamel and dentin.[51] Laser etching is painless and does not involve either vibration or heat, making this treatment attractive.[51] Furthermore, laser etching of enamel or dentin has been

reported to yield an anfractuous surface (fractured and uneven) and open dentinal tubules, both apparently ideal for adhesion.[51] The surface produced by laser etching is also acid resistant because laser irradiation of dental hard tissues modifies the calcium-to-phosphorus ratio reduces the carbonate-to-phosphate ratio and leads to formation of more stable and less acid-soluble compounds, thus reducing susceptibility to acid attack and caries.[51]

The action of lasers depends on their wavelengths and their subsequent absorbtion by the target tissue. CO_2 laser and the erbium family of lasers, (Er;Cr:YSGG and Er:YAG) are the lasers preferred for working with hard tissues like the tooth and bone, because of their absorbtion by water.[52]

Some of the laser systems have the ability to treat dental surfaces to create a rough microretentive pattern.[4] Lasers such as Nd:YAG and CO_2, have been examined, but the initial results with these lasers were not encouraging due to the thermally induced injuries to the surrounding tissues including pulpal damage.[3,10,30] Many investigators have reported the ability of the Er:YAG laser to ablate tooth structure, which has also been indicated for selective removal of carious lesion, cavity preparation and modification of dentin and enamel surfaces prior to restoring with adhesive materials.[3,10,30]

The mechanism of action of erbium lasers has been reported to be the same,[62] with only a minor difference in their wavelengths with Er:YAG being 2.94 µm as opposed to the Er;Cr:YSGG wavelength of 2.89 µm.[10,30] When the laser energy is focused onto the tooth, the water contained therein, is

heated and the steam causes an increase in the irradiated volume. This expansion surpasses the crystal strength of the dental structures, and results in ablation. This mechanism explains the anfractuous, microretentive pattern obtained after etching with the Erbium lasers.[13,19,24,30,31,41,45,51] The present in vitro study used an Er;Cr:YSGG laser to etch the enamel surface of the prepared tooth samples.

Resin based composite cements are the cements of choice for the adhesive luting of ceramic restorations.[6] Resin cements are capable of producing micromechanical attachment to the tooth structure.[20] Two types of resin cements available are dual-cured and light-cured resin cement.[27] Light-cured cements have some proven advantages in that working time is increased, the ability to remove excess cement is facilitated and this reduces the finishing time.[27] Dual-cured cements traditionally are used when ceramic thickness does not allow light penetration for maximal conversion of the luting cement.[30,43] Disadvantages of dual-cured cements include porosity from mixing, reduced working time, decreased degree of conversion and color instability due to amine degradation.[27] In accordance with the literature, the present in vitro study used a dual-cured resin cement for the bonding of ceramic blocks (5 x 5 mm) onto the surface treated tooth samples.

Earlier studies have reported on the effect of water storage on the bond strength. The International Standards Organization's report on the testing of dental materials TR110405 also states that longer periods of storage in a

solution are necessary to determine the durability of bonds.[28] Storage in water may result in hydrolytic degeneration of the interface components especially of the resin cement and/or collagen and is also detrimental to the silane-ceramic bond.[35,36] Storage in water and additional thermocycling create stress at the cementing agent/hard tissue interface.[36] In the present in vitro study the samples were stored in distilled water at 37^{o} C for a period of seven days to simulate the oral conditions.

The occlusal forces applied to a restoration may be complex and made up of a combination of forces such as shear, tension, compression and flexure.[2] The tests most widely used to examine bond strength of resin composite to dentin are tensile and shear tests.[2] Shear strength is clinically more applicable because resistance to shear stresses are important in retaining restorations that have been bonded to enamel surfaces.[50] In the present in vitro study, a conventional shear bond strength test with a crosshead speed of 0.5 mm[22] was used to evaluate the long-term durability according to ISO TR 11405:2003.

The aim of the present in vitro study was to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

A total of thirty freshly extracted maxillary central incisors were embedded in acrylic blocks using a custom-made metallic jig. After tooth preparation which extended into the enamel for 2 mm to simulate a clinical preparation for ceramic laminate veneer using a custom-made preparation

guide, the teeth were divided into three different groups namely Group A, Group B and Group C and three different surface treatments were carried out namely acid etching, laser etching and combination of acid etching followed by laser etching for Group A, Group B and Group C respectively. One sample from each group was randomly selected for a surface analysis using scanning electron microscope (SEM) analysis before bonding of ceramic blocks to the prepared teeth. A total of thirty pressed ceramic blocks were fabricated. The ceramic samples were then bonded with dual-cured resin cement to the prepared surface of the etched teeth. The bonded test samples of Groups A, B and C were subjected to aging for a period of seven days at 37^{o} C and were tested for shear bond strength using a universal testing machine.

The results were then tabulated and subjected to a statistical analysis. The mean and standard deviation were obtained for each group. One random sample from each group after the completion of shear bond strength tests was then subjected to an SEM analysis to identify the mode of failure. All photo micrographs were obtained at 10x, 500x and 1000x magnifications.

In the present study the mean shear bond strength value of acid etched enamel with 37% phosphoric acid (Group A) was 11.9470 MPa (Table I). The mean shear bond strength of laser etched enamel with Er;Cr:YSGG laser system (Group B) was 12.7640 MPa (Table II) and the mean shear bond strength of the combination of acid etching followed by laser etching (Group C) was 11.2760 MPa (Table III).

Since the mean values were different between the Groups A,B and C, One-way ANOVA statistical test was performed and it was found that there was a statistically significant difference at the 5% level of significance ($p < 0.05$) among the three groups. Following this post hoc Tukey HSD statistical analysis was performed to identify the groups with statistical significance.

On statistical comparison between the mean shear bond strengths of acid etched samples (Group A) and laser etched samples (Group B), the laser etched samples (Group B) recorded higher mean bond strength than the acid etched samples (Group A) but there was no statistically significant difference between the groups observed.

On statistical comparison between the mean shear bond strengths of acid etched samples (Group A) and combined acid etched and laser etched samples (Group C), acid etched samples (Group A) exhibited higher mean shear bond strengths than the samples which were treated with combined use of acid etching followed by laser etching (Group C). However no statistically significant difference was observed between the groups.

On statistical comparison between the mean shear bond strengths of laser etched samples (Group B) and combined acid etched and laser etched samples (Group C), laser etched samples (Group B) exhibited higher mean shear bond strength than the samples treated using a combination of acid etching followed by laser etching (Group C). The difference observed between

the groups was statistically significant, at the level of significance 5% ($p < 0.05$).

The effect of laser etching, acid etching and combination of acid etching and laser etching on enamel has been evaluated in various researches.[4,19,30,32,50,51,53] The shear bond strength of composites,[4,30,32,50,53] and ceramics[19] to the treated enamel surface have been evaluated in literature. Previous studies show that the bond strength values achieved with laser etching of enamel have been comparable to the bond strength values achieved with acid etching.[13,19,30,32,50,51,53] Application of laser etching has been suggested as an alternative to acid etching considering its etching property and other advantages like the increased resistance to caries, ease of handling and faster means of etching. The results obtained with the present study are broadly in line with the results obtained in the previous study. However, researches identical to the present study parameters are sparse in the literature.

Dundar et al (2009)[24] had comparatively evaluated the shear bond strength of ceramic to enamel after different surface treatment of enamel (acid etching, laser etching and a combination of acid etching and laser etching). The mean shear bond strength value obtained with acid etching (15.44 MPa) was slightly higher than that achieved with laser etching (12.89 MPa) and the combination of acid etching followed by laser etching (13.87 MPa) but the results were not statistically significant. The compositions of the ceramic material and the resin cement used for bonding to the enamel surface in this

study are different from the ceramic and resin cement used in the present study. But the shear bond strength values yielded by the etching methods are comparable with the shear bond strengths obtained in the present study.

The results obtained in the present study are in accordance with the study by Visuri et al (1996)[53] which revealed higher shear bond strength value of composite resin when it was bonded to laser prepared tooth surface (12.9 MPA) than with acid etched tooth surface (7.3 MPa).

Lin et al (1999)[30] stated that the use of an Er;Cr:YSGG laser provided surfaces that are receptive to attachment of restorative materials. Enamel surfaces treated with the Er;Cr:YSGG laser (23.7 MPa) yielded shear bond strengths similar to those obtained with acid etched bur-cut enamel (23.3 MPa) and the author has suggested the use of laser.

Usumez et al (2003)[51] reported that the microtensile bond strength of porcelain laminate veneers bonded to tooth surfaces that were laser etched (12.1 MPa) showed results similar to acid etched (13 MPa) tooth surfaces.

Moslemi et al (2010)[41] stated that there was no statistically significant difference between shear bond strength values obtained with acid (37% phosphoric acid) etching (23.51 MPa) and combination laser and acid etching (21.44 MPa) and these results are in accordance with the present study.

In the present in vitro study the qualitative analysis of the treated surface of Group A sample before bonding to ceramic block showed a definite, type III key-hole pattern throughout the surface. The surface

presented a uniform micro-retentive pattern over the entire etched area. No smear layer was visible over the etched surface. The surface analysis of the Group B sample before bonding to ceramic block showed no definite or uniform pattern and an absence of a smear layer. The surface had numerous voids and a definite micro-retentive topography with several raised elevations and depressions. The surface analysis of the Group C sample before bonding to ceramic block revealed a heterogeneous topography, showing both acid induced porosities and laser induced surface roughness.

The qualitative analysis of the surface of Group A sample after debonding revealed a mixed failure pattern. There was a predominant cohesive failure occurring within the cement and an adhesive pattern of fracture between the enamel and cement. The surface analysis of the Group B sample after debonding revealed a predominantly cohesive mode of failure in the cement with few areas of adhesive failure. The surface analysis of the Group C sample after debonding revealed a predominantly adhesive failure pattern.

The scanning electron microscope (SEM) analysis findings are in correlation with the results obtained from the shear bond strength test. The higher shear bond strength values obtained with Group B and Group A test samples could be attributed to the predominantly cohesive nature of failure with these groups. The lower percentage of adhesive failure areas for Group B test sample as compared to the Group A test sample could account for its higher shear bond strength value obtained in the present study.

The predominantly adhesive mode of failure of Group C test sample is in correlation with the significantly lower shear bond strength values obtained with this group.

The results obtained from the qualitative analysis of this study are in correlation with the results obtained from the quantitative analysis of the study with Group B (laser etching) samples showing the highest mean shear bond strength followed by Group A (acid etching) and Group C (combination of acid etching followed by laser etching) samples.

The present in vitro study was conducted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods. Although this study reports a higher mean shear bond strength for laser etched enamel as opposed to acid etched enamel there was no statistical significance. A larger sample size might be more indicative of definite predictable results. This study also reports higher mean shear bond strength for laser etched samples on comparison with samples which received a combination of acid etching followed by laser etching. The difference observed between laser etching and a combination of acid etching and laser etching was statistically significant. The lower mean shear bond strengths exhibited by the samples which were treated by a combination of acid etching followed by laser etching may be due to the lower amount of surface roughness produced by it as observed under the SEM. It is well accepted that a high surface roughness is closely related to greater bonding.[24]

Acid etching with 37% phosphoric acid has yielded satisfactory bond strengths for a long time, but it's liability to leave the enamel demineralised has always been a point of concern. Laser etching, in turn leaves the enamel more resistant to acid dissolution.[28,31] The area to be etched can be very precisely limited with a laser without any damage to the surrounding tooth structure. The results of the present study suggest laser etching to be a viable alternative to acid etching.

Future studies need to be done to evaluate the long term shear bond strength after laser etching and also studies evaluating the effect of thermo-cycling on shear bond strength would better predict the in vivo outcome of laser etching. This in vitro study limited the depth of preparation into the enamel, future studies may also look into the effect of extending the preparation depth into the dentin. Further studies may also be done to evaluate the effect of different power settings of the laser on the bond strength.

CONCLUSION

The following conclusions were drawn from this present in vitro study, which was conducted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods:

1. The mean shear bond strength of the bond between ceramic and enamel pretreated with 37% phosphoric acid etching (Group A) was **11.9470 MPa**.
2. The mean shear bond strength of the bond between ceramic and enamel pretreated with Er;Cr:YSGG laser etching (Group B) was **12.7640 MPa**.
3. The mean shear bond strength of the bond between ceramic and enamel pretreated with a combination of 37% phosphoric acid etching followed by Er;Cr:YSGG laser etching. between enamel surface and porcelain after combined use of etching of the tooth surface with 37% phosphoric acid and with Er;Cr:YSGG laser system (Group C) was **11.2760 MPa**.
4. On overall comparison, the mean shear bond strength values of the three test groups, namely,
    - ❖ Group B (laser etching with Er;Cr:YSGG laser system): highest mean shear bond strength
    - ❖ Group A (acid etching with 37% phosphoric acid): moderate mean shear bond strength

- ❖ Group C (acid etching with 37% phosphoric acid followed by laser etching with Er;Cr:YSGG laser system): least mean shear bond strength

Group B (12.7640 MPa) > Group A (11.9470 MPa) > Group C (11.2760 MPa)

Statistical significance was shown among the three groups.

5. On comparison, the mean shear bond strength between ceramic and enamel surface after etching with 37% phosphoric acid (Group A, mean value-11.9470 MPa) was lower than the mean shear bond strength after etching with Er;Cr:YSGG laser system (Group B, mean value-12.7640 MPa) but the difference was not statistically significant.
6. On comparison, the mean shear bond strength between ceramic and enamel surface after etching with 37% phosphoric acid (Group A, mean value-11.9470 MPa) was higher than the mean shear bond strength after combination of etching with 37% phosphoric acid followed by etching with Er;Cr:YSGG laser system (Group C, mean value-11.2760 MPa) but the difference was not statistically significant.
7. On comparison, the mean shear bond strength between ceramic and enamel surface after etching with an Er;Cr:YSGG laser system (Group B, mean value-12.7640 MPa) was higher than the mean shear bond strength after combination of etching with 37% phosphoric acid followed by etching with Er;Cr:YSGG laser system (Group C, mean

value-11.2760 MPa) and the difference was statistically significant (p-value < 0.05) (Group B > Group C).

8. Qualitative analysis of the enamel surface before ceramic bonding by scanning electron microscopy (SEM) after etching with 37% phosphoric acid revealed the presence of definite enamel key-hole pattern throughout the surface and a uniform, type III micro-retentive pattern throughout the etched area and devoid of smear layer.
9. Qualitative analysis of the enamel surface before ceramic bonding by scanning electron microscopy (SEM) after etching with Er;Cr:YSGG laser system exhibited an absence of smear layer, irregular micro-retentive surface with prominent elevations and depressions of varying degrees throughout the surface.
10. Qualitative analysis of the enamel surface before ceramic bonding by scanning electron microscopy (SEM) after a combination etching with 37% phosphoric acid followed by Er;Cr:YSGG laser system revealed a heterogeneous surface topography with shallow, irregular, laser-ablated surface elevations and depressions along with acid induced porosities and devoid of smear layer.
11. Qualitative evaluation of the mode of failure of debonded test sample of ceramic bonded to enamel etched with 37% phosphoric acid exhibited a mixed adhesive and cohesive failure with a predominantly cohesive failure pattern within the resin cement as observed on the tested sample.

12. Qualitative evaluation of the mode of failure of debonded test sample of ceramic bonded to enamel etched with Er;Cr:YSGG laser system revealed a predominantly cohesive pattern in the resin cement with few areas of adhesive failure at the enamel-cement interface as observed on the tested sample.
13. Qualitative evaluation of the mode of failure of debonded test sample of ceramic bonded to enamel etched with a combination of 37% phosphoric acid followed by Er;Cr:YSGG laser etching revealed a predominantly adhesive failure at the enamel-cement interface as observed on the tested sample.

SUMMARY

The present in vitro study was conducted to comparatively evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching methods.

A total of thirty three freshly extracted teeth were embedded in acrylic blocks using a custom-made jig. The teeth were prepared using a custom-made preparation guide limiting the depth of the preparation into the enamel. The prepared teeth were divided into three different groups, as Group A, Group B and Group C and were subjected to three different surface treatments, namely, acid etching, laser etching and combination of acid etching followed by laser etching for Group A, Group B and Group C respectively. One sample from each surface treated group was randomly selected for a surface analysis using scanning electron microscope (SEM) analysis before bonding of ceramic blocks to the prepared teeth. A total of thirty ceramic blocks were fabricated and were then bonded to the teeth etched with their respective surface treatment methods. The bonded test samples of Groups A, B and C were subjected to aging for a period of seven days and were tested for shear bond strength using a universal testing machine. One debonded test sample from Groups A, B and C was randomly selected for a qualitative analysis by SEM analysis. The results were tabulated and subjected to statistical analysis.

Statistical analysis revealed that Group B (laser etching) exhibited the highest mean shear bond strength value followed by Group A (acid etching) and Group C (combination of acid etching followed by laser etching). The difference in the shear bond strength values among the three groups was statistically significant as was found using One-way ANOVA. (Group B > Group A > Group C)

Tukey HSD post hoc comparisons between the test groups revealed a statistically insignificant difference in mean shear bond strength value for Group A in comparison to Groups B and C and a statistically significant difference in mean shear bond strength value for Group B in comparison to Group C.

The qualitative analysis by scanning electron microscope (SEM) before bonding of ceramic to the enamel of the prepared tooth after laser etching surface treatment (Group B) had exhibited greater surface irregularities compared to acid etching (Group A) and combination of acid etching followed by laser etching (Group C)

The qualitative analysis by scanning electron microscope (SEM) of debonded test samples had revealed primarily cohesive failure with more residual cement on tooth surface in Group B sample, primarily cohesive failure with less residual cement particles on Group A sample, whereas Group C sample had exhibited primarily adhesive failure.

The results of the qualitative analysis of the test samples are in correlation with the quantitative analysis of the test samples in this study.

The results of the present in vitro study revealed that there was no statistically significant difference in the mean shear bond strength values between laser etched and acid etched test groups, but a significant difference between the laser etched group and the test group with combination of etching with acid followed by laser was found. Although acid etching is a widely used method, the advantages and superior technology of laser etching makes it a predictable alternative for bonding porcelain to enamel surfaces,[9,18,50] as has been revealed from the present results.

Future studies need to be done to evaluate the long term shear bond strength after laser etching and also studies evaluating the effect of thermo-cycling on shear bond strength would better predict the in vivo outcome of laser etching. This in vitro study limited the depth of preparation into the enamel; future studies may also look into the effect of extending the preparation depth into the dentin. Further studies may also be done to evaluate the effect of different power settings of the laser on the bond strength to enhance the results obtained with the present study.

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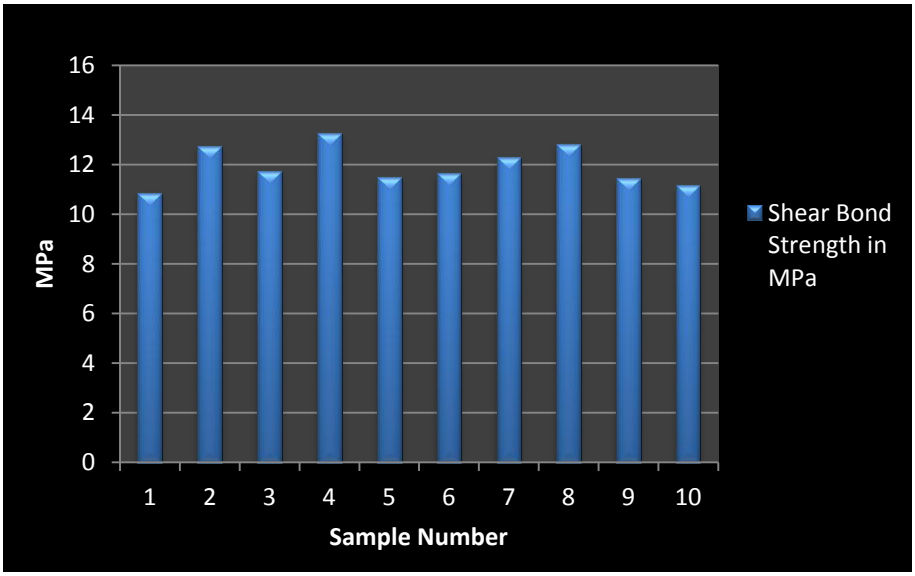
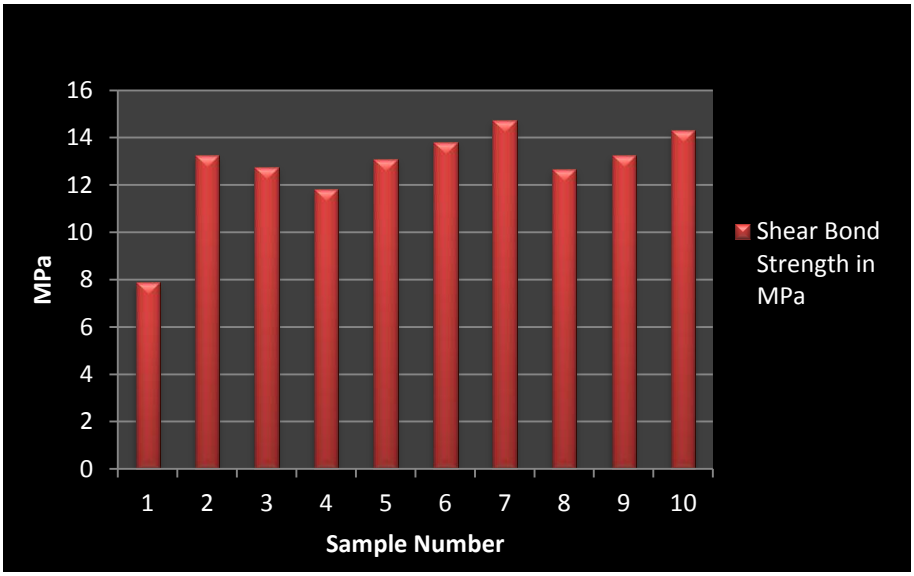
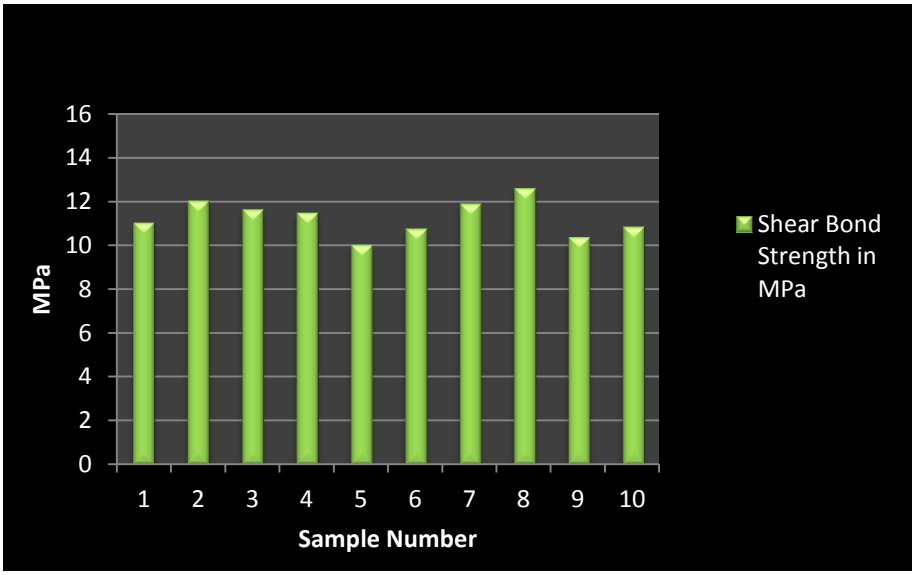
Graph I: Basic values of shear bond strength of Group A (acid etching) test samples



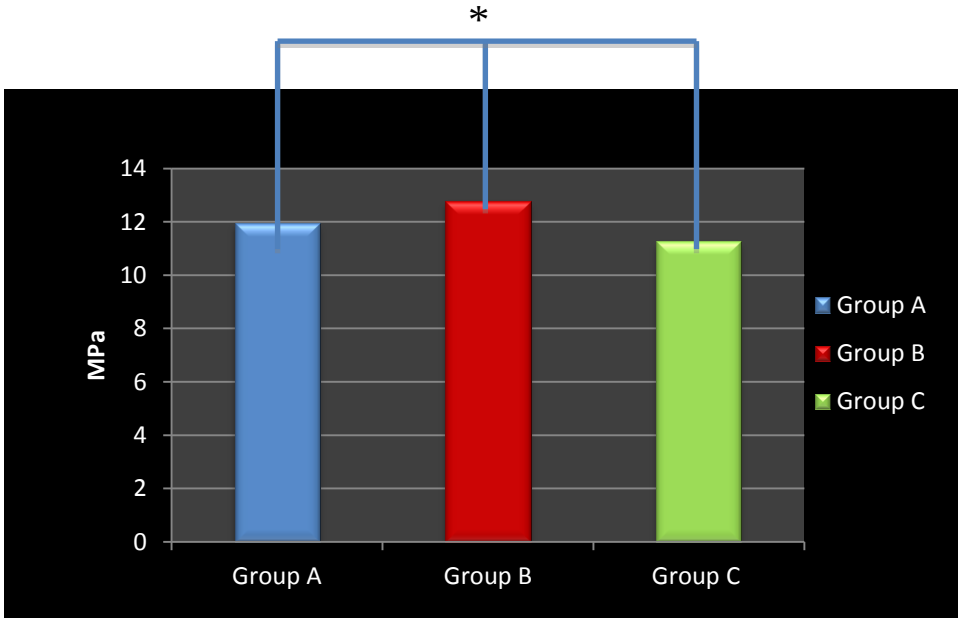
Graph II: Basic values of shear bond strength of Group B (laser etching) test samples



Graph III: Basic values of shear bond strength of Group C (combination of acid etching followed by laser etching) test samples

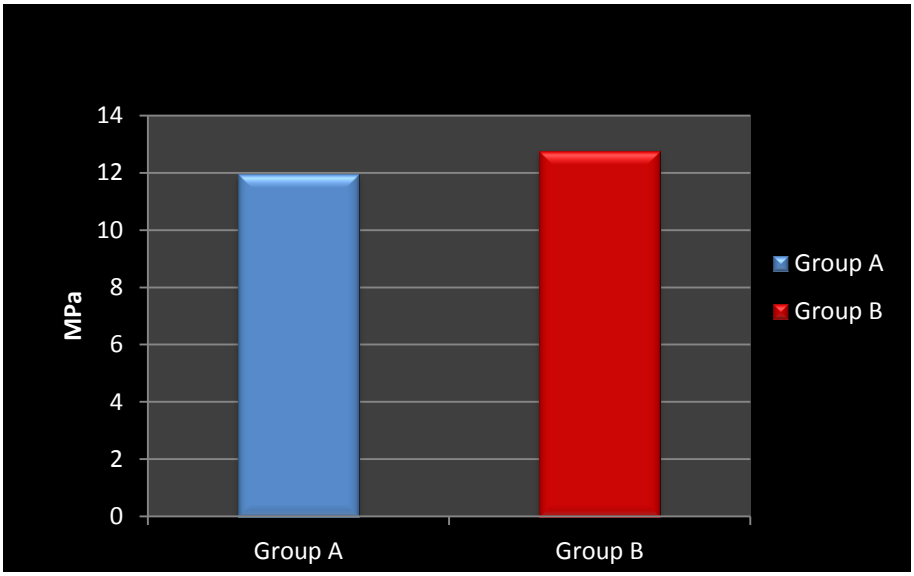


Graph IV: Comparison of mean shear bond strength values of Group A (acid etching), Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples

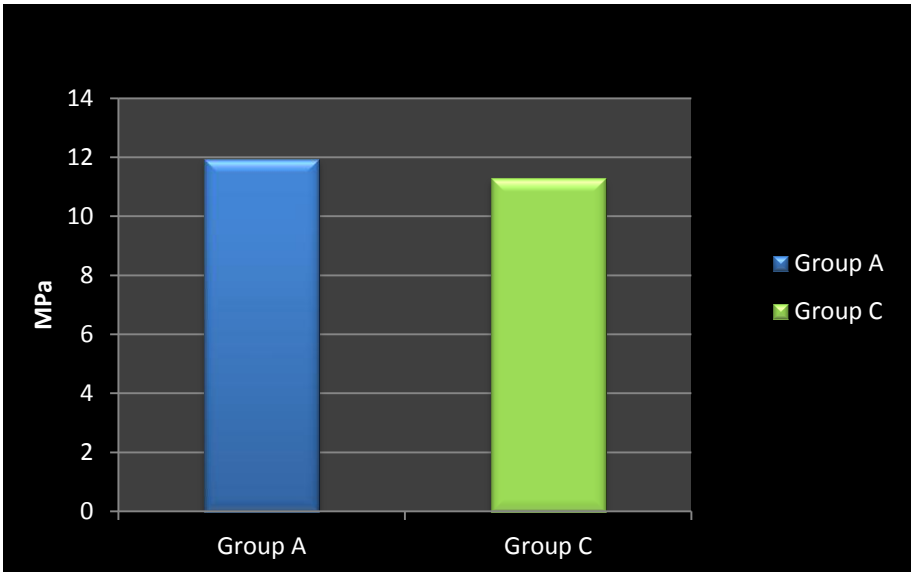


*** Significant at 5% level**

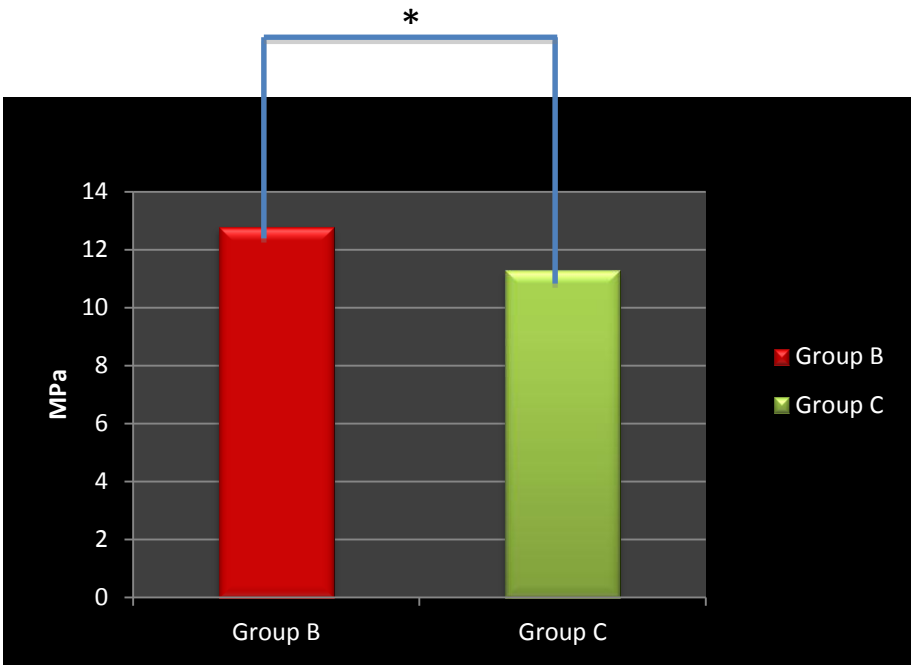
Graph V: Comparison of mean shear bond strength values of Group A (acid etching) and B (laser etching) test samples



Graph VI: Comparison of mean shear bond strength values of Group A (acid etching) and C (combination of acid etching followed by laser etching) test samples



Graph VII Comparison of mean shear bond strength values of Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples



*** Significant at 5% level**

MATERIALS AND EQUIPMENTS

Fig.1: Recently extracted maxillary central incisors

Fig.2: Separating discs

Fig.3: Autopolymerizing clear acrylic resin

Fig.4: Die lubricant

Fig.5: Inlay wax

Fig.6: Sprue wax

Fig.7: Investment ring and crucible former

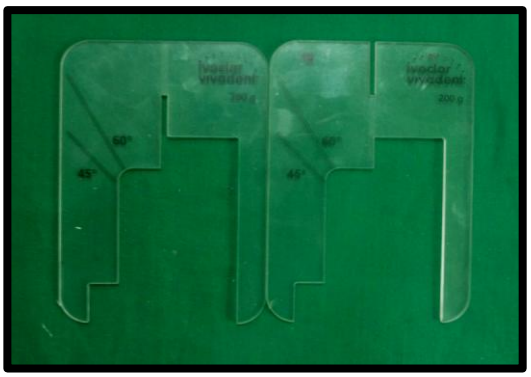


Fig.8: Pattern sprue guide

Fig.9: Phosphate bonded investment material

Fig.10: Colloidal silica

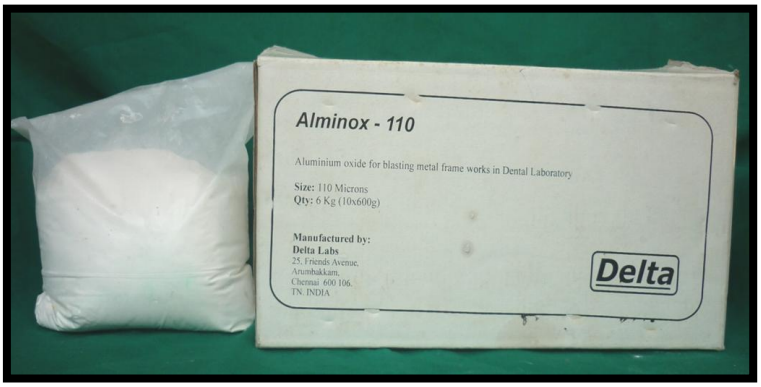


Fig.11: Aluminium oxide powder

Fig.12: Diamond disc

Fig.13: Silicon carbide impregnated burs coarse

Fig.14: Silicon carbide impregnated burs fine

Fig.15a: Primer, b: Adhesive, c: Bonding agent, d: Silane coupling agent

Fig.16: Dual-cure resin luting cement

Fig.17: P.K. Thomas wax up instruments

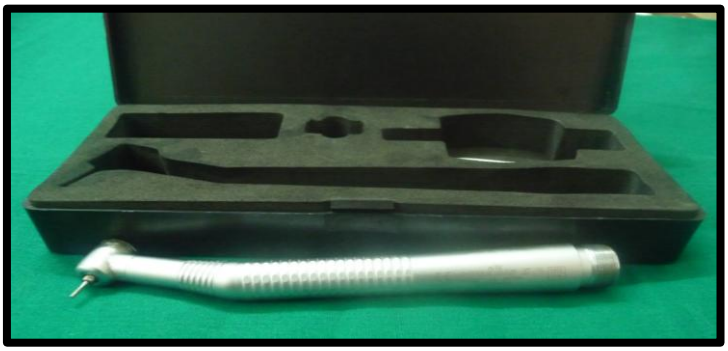


Fig.18: Aerotor hand piece

Fig.19: Inverted cone diamond abrasive

Fig.20: Flat end tapered diamond abrasive

Fig.21: Light cure unit

Fig.22: Vacuum mixer

Fig.23: Burnout furnace

Fig.24: Sandblaster

Fig.25: Incubator

Fig.26: Universal testing machine

Fig.27: Scanning electron microscope

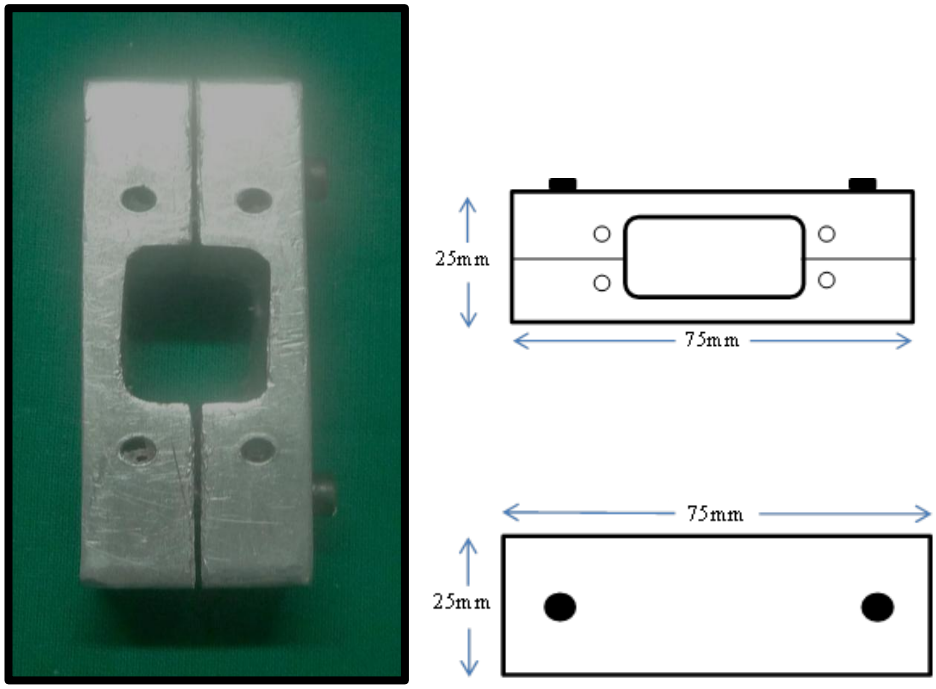


Fig.28: Custom-made stainless steel split mounting jig

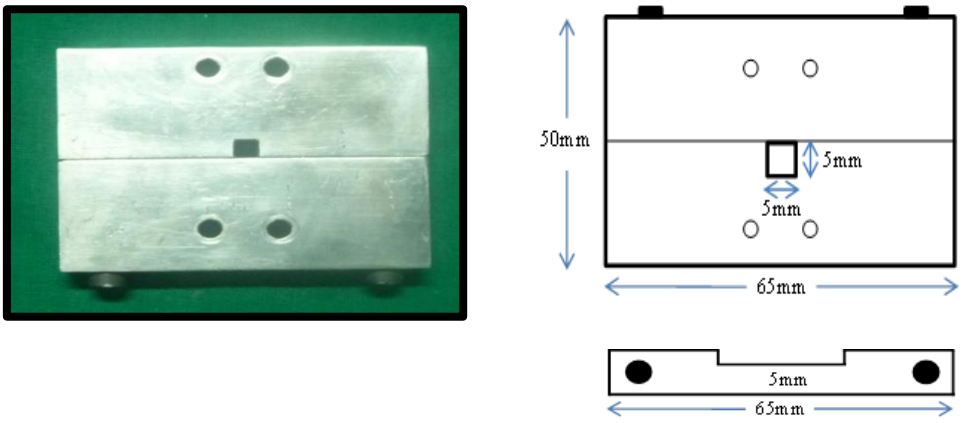


Fig.29: Custom-made stainless steel tooth preparation guide

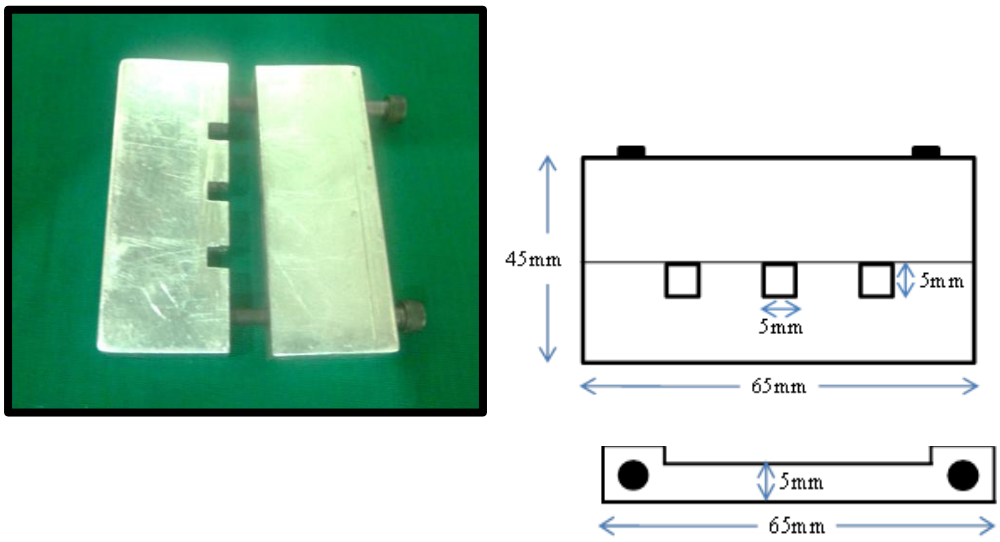


Fig.30: Custom-made stainless steel split mold

Fig.31: Lithium disilicate ingots

Fig.32a: Boron nitride, b: Plunger

Fig.33: Ceramic press furnace

Fig.34: 7% Hydrofluoric acid gel

Fig.35: 37% Phosphoric acid

Fig.36: Er;Cr:YSGG laser system

METHODOLOGY

Selection of teeth

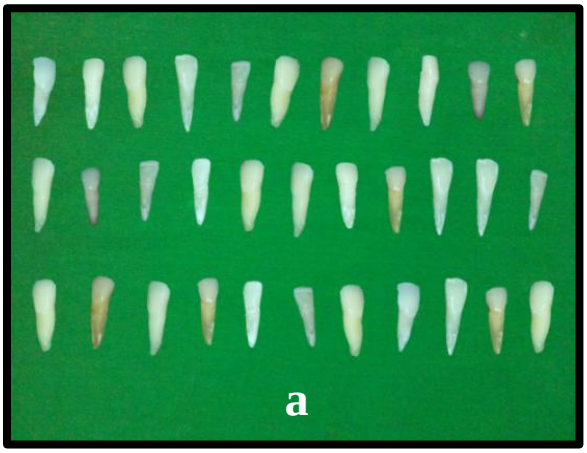


Fig.37a: Selected teeth

b: Cleaned tooth

c: Sectioned tooth

Placement of teeth in custom-made jig

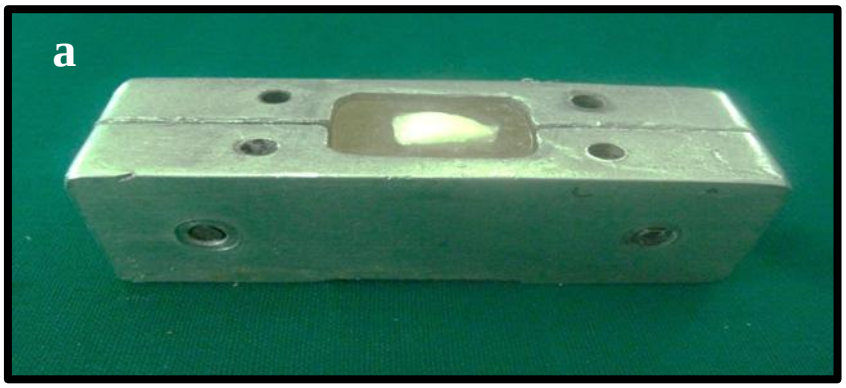


Fig.38a: Mold filled with acrylic resin and tooth placed
b: Finished sample

Preparation of teeth

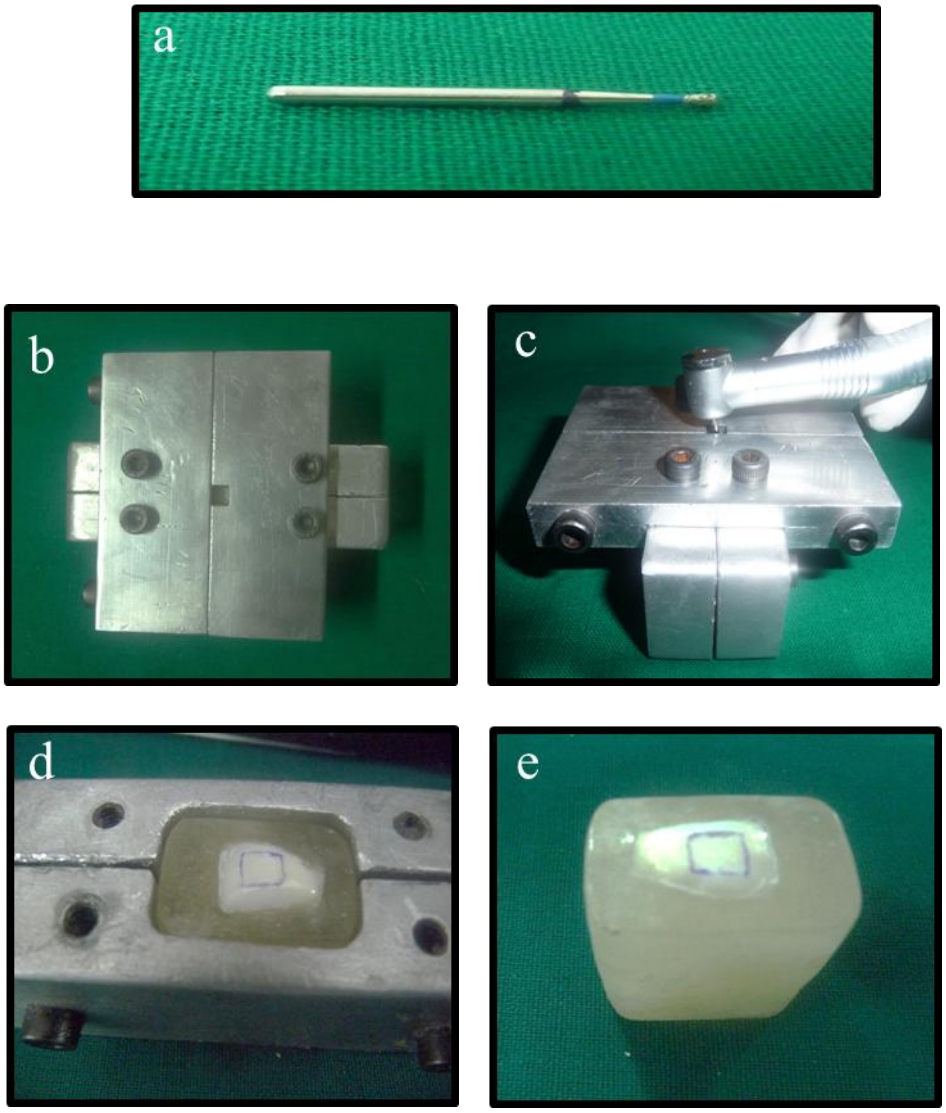


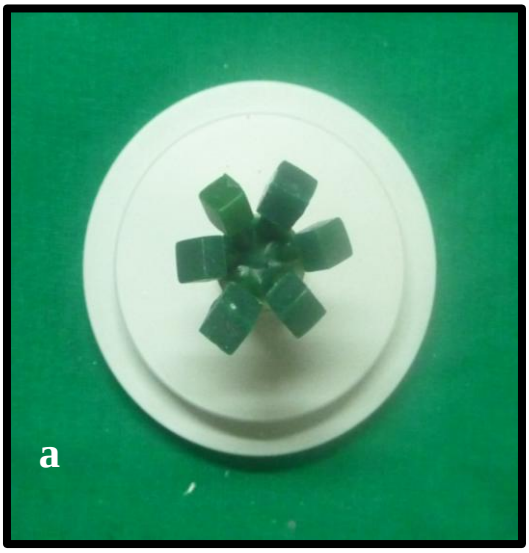
Fig.39a: Premarked bur
b: Tooth preparation guide secured in place over the jig
c: Tooth preparation done upto the marking on the bur
d: Completed preparation
e: Marked surface outlining completed preparation

Fabrication of ceramic blocks

Preparation of wax blocks

Fig.40a: Custom made split mold placed against flat glass plate
b: Mold space filled with inlay wax
c: Completed wax blocks

Spruing of wax blocks



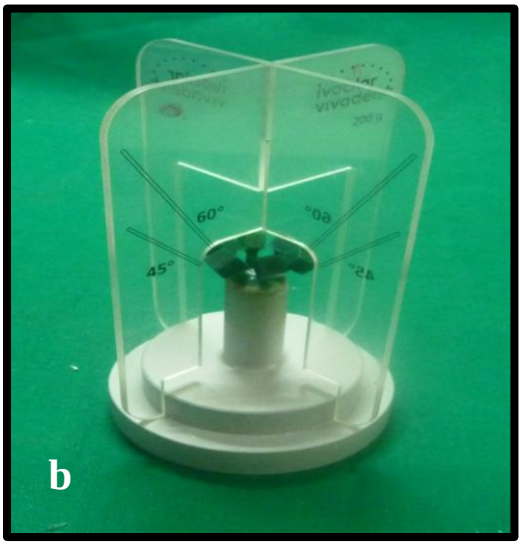


Fig.41a: Wax blocks attached to crucible former
b: Angle verification using pattern sprue guide

Investing the wax blocks

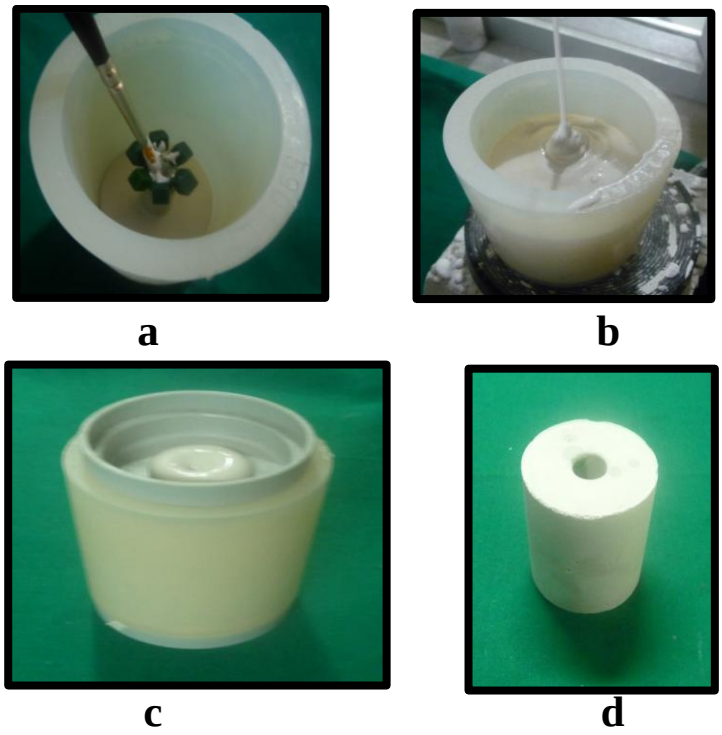


Fig.42a: Investment being gently applied on with a brush
b: Investment gently vibrated
c: Excess investment material being removed
d: Set investment mold

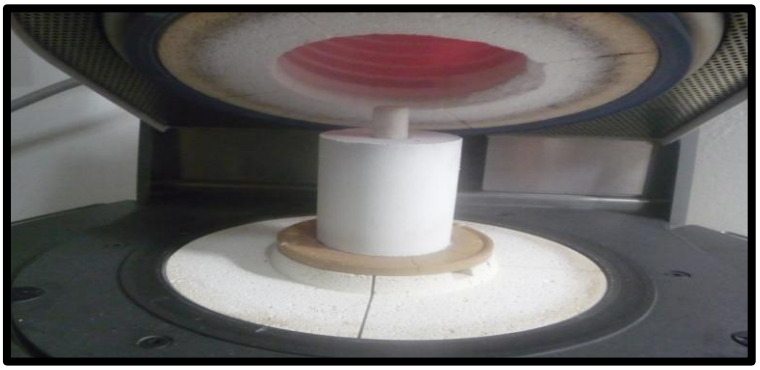
Burnout procedure for wax blocks

Fig.43: Burnout furnace

Pressing of ceramic

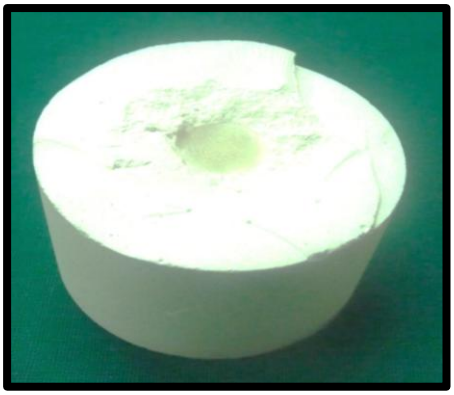
a

b



c

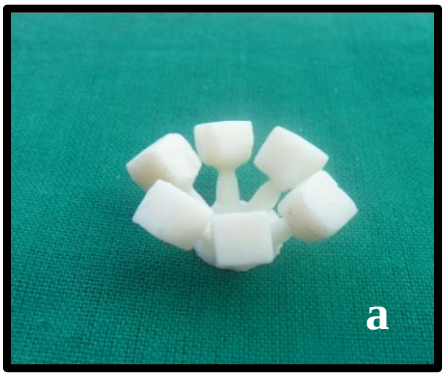
d

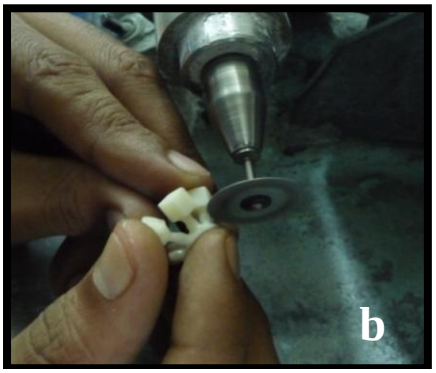


e

Fig.44a: Placement of ingot
b: Placement of plunger
c: Investment mold placed in press furnace
d: Investment mold on completion of pressing
e: Sectioned investment mold for divesting

Divesting of ceramic blocks





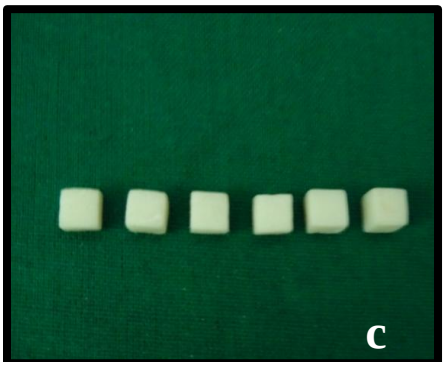


Fig.45a: Divested mold
b: Sectioning of sprues
c: Finished ceramic blocks

Preparation of ceramic blocks for bonding

Fig. 46: Ceramic blocks etched with 7% hydrofluoric acid gel

Etching of prepared teeth surfaces

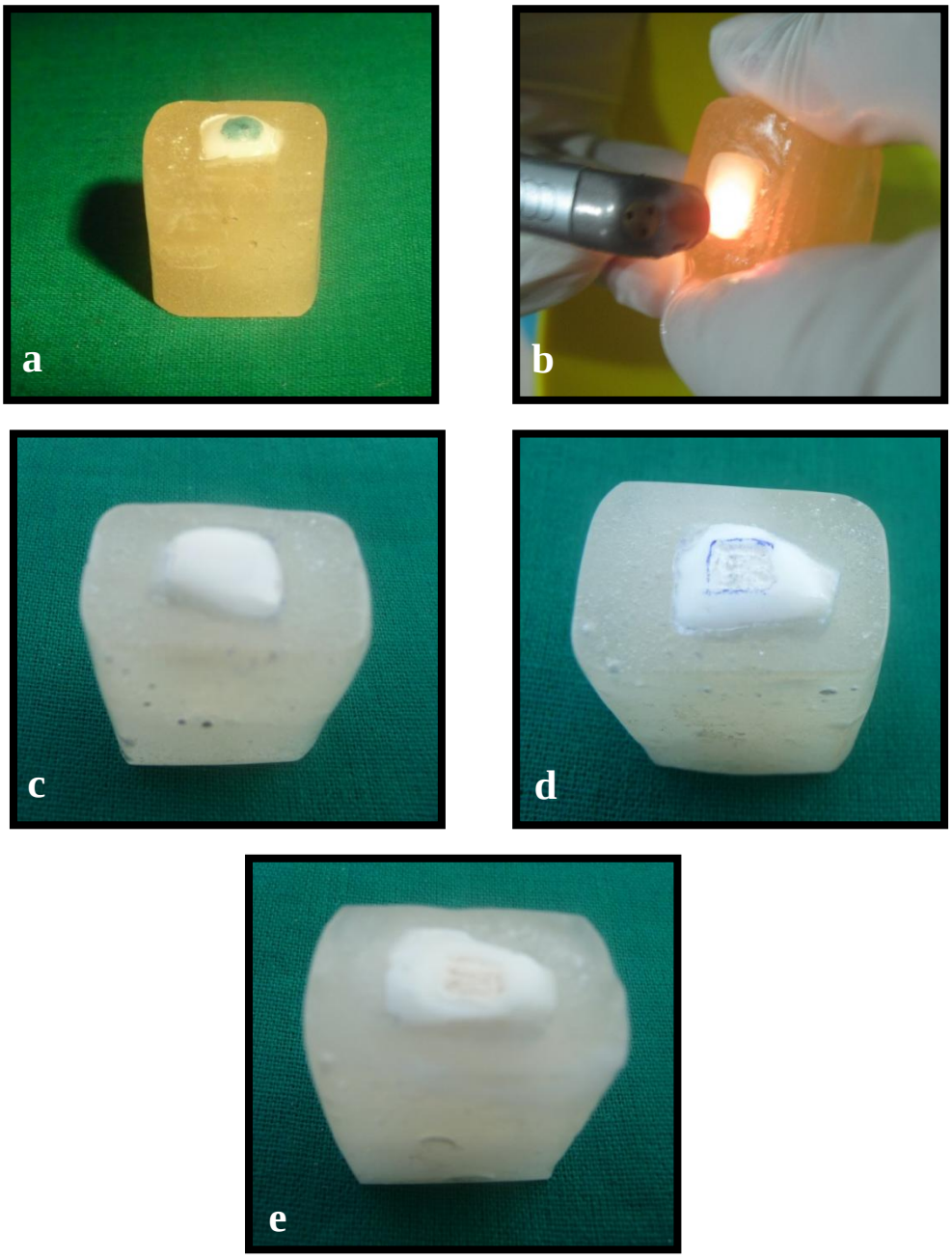


Fig.47a: Etching with phosphoric acid
b: Laser etching
c: Tooth surface after acid etching
d: Tooth surface after laser etching
e: Tooth surface after combination acid etching followed by laser etching

Cementation of ceramic blocks

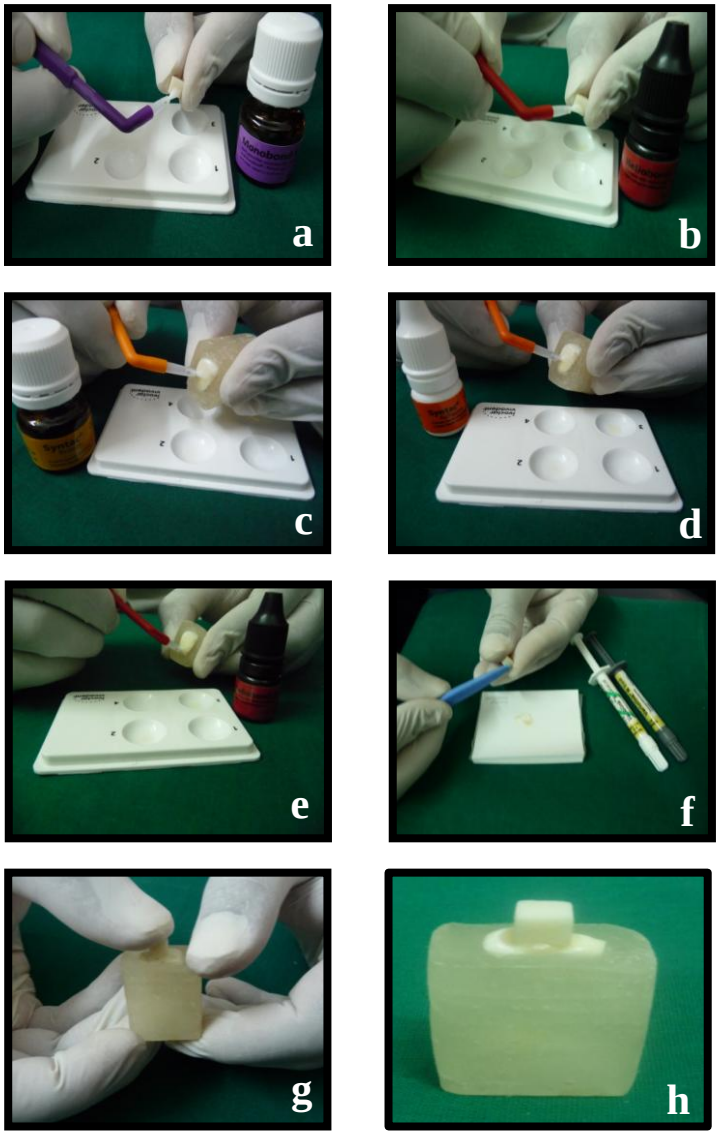
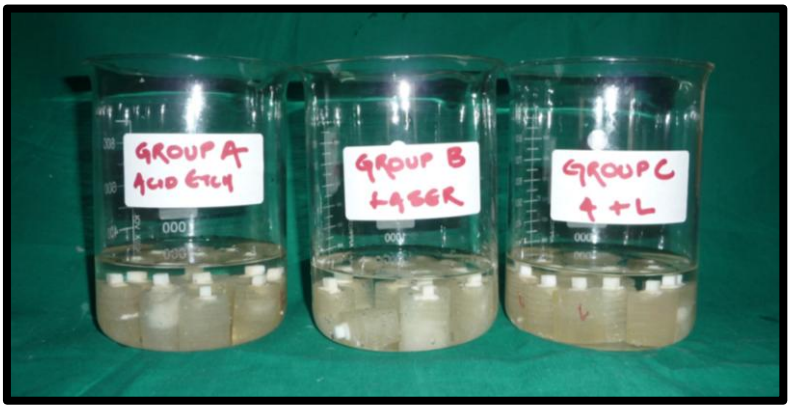
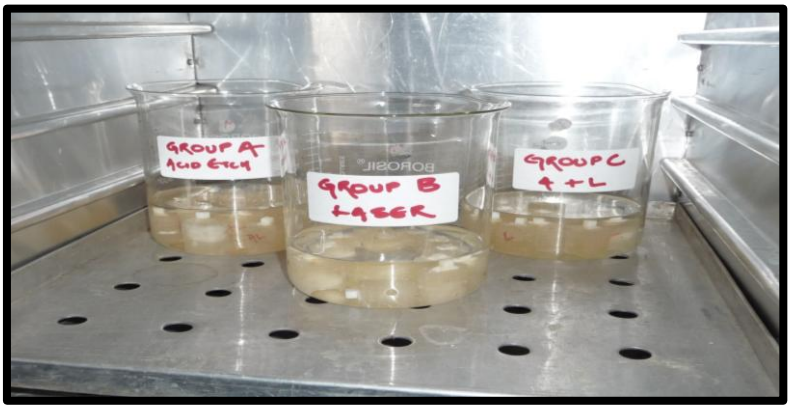


Fig.48a: Application of the silane coupling agent to the ceramic block
b: Application of the bonding agent to the ceramic block
c: Application of primer to the tooth surface
d: Application of adhesive to the tooth surface
e: Application of bonding agent to the tooth surface
f: Dual cure resin cement mixed and applied to the ceramic block
g: Ceramic block pressed against the tooth under light finger pressure
h: Ceramic block bonded to surface treated tooth test sample

Aging of test samples



a



b

Fig.49a: Test samples stored in water kept for aging

b: Test samples kept in an incubator

Shear bond strength test of test samples

Fig.50: Test sample undergoing shear bond strength test using universal testing machine

Fig.51: Debonded test samples

Qualitative analysis of the test samples

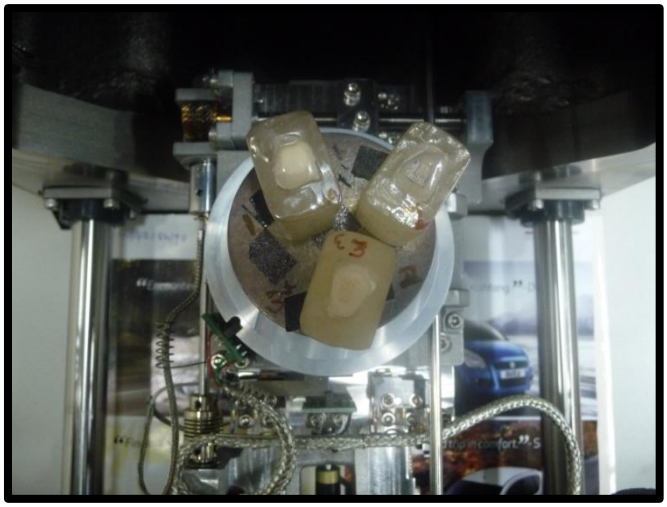


Fig.52: SEM analysis of the prepared teeth samples before bonding with ceramic blocks

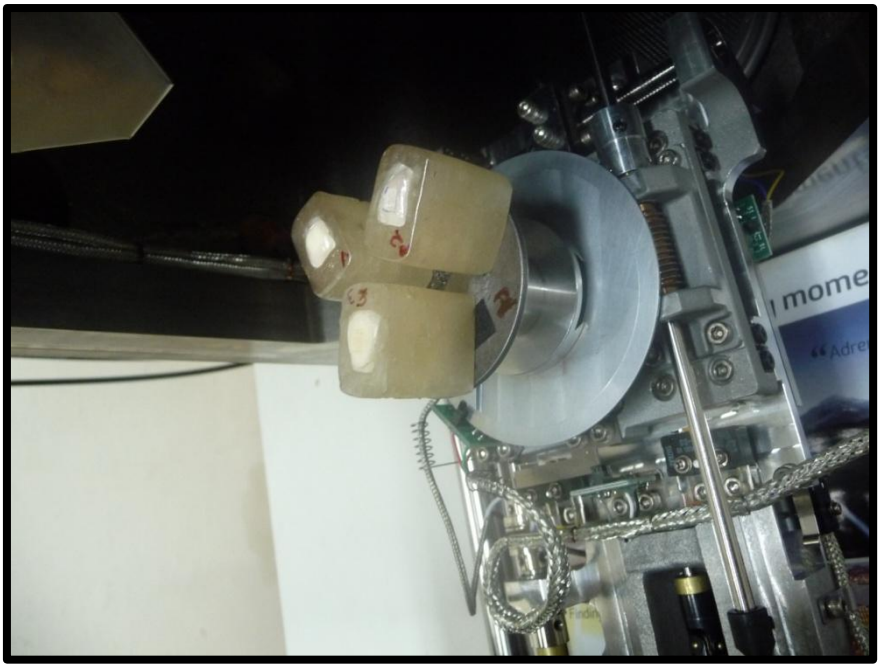


Fig.53: SEM of cemented test samples after debonding